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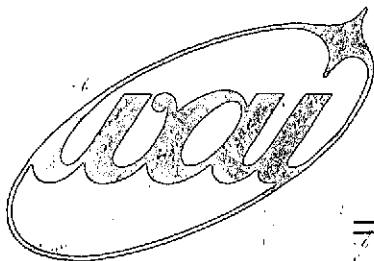
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SCIENTIFIC, ENGINEERING & MANAGEMENT SERVICES

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WAI-101  
FEASIBILITY STUDY OF A HIGH TEMPERATURE  
RADIATION FURNACE FOR  
SPACE APPLICATIONS  
FINAL REPORT

April 1973

Submitted to:

George C. Marshall Space Flight Center  
National Aeronautics & Space Administration  
Marshall Space Flight Center, Alabama 35812

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## A B S T R A C T

This document is the final report for the National Aeronautics and Space Administration, Marshall Space Flight Center, Contract NAS8-28059. The study has investigated the feasibility of a high temperature general purpose furnace for use in space. It was determined that no commercial furnaces exist which could, even with extensive modifications, meet the goals of temperature, power, weight, volume, and versatility originally specified in the contract Statement of Work. A feasible furnace design which does substantially meet these goals while employing many of the advanced features of the commercial furnaces is developed and presented.

### A C K N O W L E D G E M E N T

In the course of conducting this study, valuable assistance and guidance were provided by Marshall Space Flight Center (MSFC) personnel. Mr. A. Q. Hudgins, S&E-PT-PDE, administered and monitored the program, while Messrs. H. F. Wuenscher, S&E-PE-DIR, and L. Burge, S&E-PT-A established basic technical guidelines.

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## SUMMARY

This summary report describes the conceptual design of a high temperature radiation furnace suitable for general materials and manufacturing experiments in a space environment. The work was performed by Weiner Associates, Inc. (WAI), under Marshall Space Flight Center (MSFC) Contract NAS8-28059. The concept was evolved after it became evident that no contemporary commercial furnaces exist which can adequately fulfill design and versatility that are compatible with space usage. Nevertheless, extensive use of state-of-the-art components, and techniques derived from these furnaces is made.

The furnace concept offered is a single general purpose facility that is designed to operate at 2300°C with a 5 Kg sample in oxidizing, reducing, inert, or vacuum environments. It requires 6 Kw maximum power and a heat removal system with a capacity of 4.5 Kw. Total furnace weight is estimated at 29 Kg (excluding the heater power and heat removal control systems and sample).

The furnace is based upon a modularized design to allow rapid maintenance of parts and component and/or subsystem and sample replacement. All items required for a specific experiment (i.e., crystal growing, directional solidification, etc.) are mounted in the furnace door which is easily removed and replaced by alternate experiment/door combinations. The heaters, insulation, pressure hull and cooling jacket comprise an operational unit suitable for all types of experiments. Changing experiments requires, at most, only changing the door assembly. Other components will remain in place and can be part of the basic space vehicle structure.

## 1.0 INTRODUCTION

The advantages of materials processing in a zero-gravity environment have been expressed for many years. Thermal convection, crucible contamination, and density segregation have limited the development of exotic and high purity alloys and fiber-reinforced composites. Low energy reactions (e.g. crystal growth) are sensitive to gravity influences. It is not surprising that some of the first experiments proposed for manned space flights were of this type.

Results of the Apollo 14 composite casting demonstration (Ref. 1) indicate that a significant advancement in materials processing techniques can be obtained in the absence of gravity. These tests also indicate that a major development of zero-gravity handling and processing technique is necessary.

The limited Apollo 14 experiments did not adequately answer many of the questions concerning the behavior of macroscopic materials and reactions when not subject to gravity forces. The maximum sample size (1.74cm dia. x 7.5cm long), temperature range ( $<100^{\circ}\text{C}$ ) and limited sample handling capability of the Apollo Furnace and limitations on allowable experiment time during trans-Lunar and trans-Earth coast periods indicate the need for a more capable furnace and more compatible mission.

The Shuttle and Sortie missions during the next few years constitutes an ideal platform for zero-gravity experiments. For the first time, adequate power, space, and time will be available for truly extensive experimentation. Eventually, with the establishment of permanent space stations, experimenters and possibly manufacturers will be able to use a zero-gravity furnace facility to its fullest capabilities.

This investigation was initiated in order to determine the feasibility of such a general purpose furnace. A review of contemporary commercially available furnaces was accomplished. When it became apparent that no commercial furnaces were suitable, even with extensive modifications, a conceptual design study was completed. The results of these investigations are included in this report.

It must be stressed that the designs presented here are conceptual in nature. This is entirely consistent with the original contract Statement of Work. Detail design of a general purpose space furnace must be predicated by decisions as to the relative importance of the initial program goals and definition of the exact interface and mission requirements.

Section 2 presents a survey of commercially available high temperature furnaces. Section 3 describes earlier designs of zero-gravity furnaces. Section 4 presents the furnace design concept which resulted from this study. Section 5 discusses the considerations that have established this concept. The technical analyses performed to support the feasibility of the concept are presented in separate appendices (Appendix A - Material Thermal Properties; Appendix B - Material Structural Properties; Appendix C - Structural Analysis; Appendix D - Thermal Analysis; and, Appendix E - Components).

## 2.0 COMMERCIAL EQUIPMENT

A survey was made to determine whether commercially available high temperature furnaces would, with suitable modifications, meet the requirements established in the program objectives. A further purpose was to determine whether specific components of one or more commercial furnaces could be incorporated into a compositely designed multi-purpose high temperature space furnace. The survey included some fourteen (14) firms who are the major high temperature furnace manufacturers in the country.

It was recognized at the outset of this survey that most commercial high temperature furnaces are designed for one, or at most a limited number of operations and that the desired multi-purpose capability would not be available in one furnace.

Table 2.1 is a sampling of some of the characteristics of furnaces manufactured by commercial suppliers. All manufacturers make a variety of general purpose high temperature furnaces and some produce special purpose furnaces (i.e. single crystal growth, directional solidification, etc.). The listing in Table 2.1 is confined to resistance heated radiation furnaces primarily of the cold wall vacuum variety. Oxidizing environment furnaces were also added to the listing.

The vacuum furnaces typically employ tungsten mesh, tantalum sheet or molybdenum sheet heaters (depending on the intended maximum temperature). Insulation is usually provided by a series of concentric radiation shields around the heating element and a similar stack of shields at the top and bottom. This assembly is installed in a water cooled steel vacuum chamber or, less frequently, in a bell jar system. Relatively large low voltage/high current power supplies are usually required.

The Thermo-Electron Corporation furnace offers the most efficient utilization of power having the largest working volume and the smallest power consumption of any of the furnaces listed. This is accomplished by the use of an insulation system which consists of large numbers of concentrically wound foils separated from one another by a sparse coating of a low thermal conductivity ceramic. This multi-foil insulation is claimed to reduce heat losses and power requirements by 85 to 90 percent. A correspondingly large reduction in cooling water requirements is also achieved.

Little data were obtained on the various manipulative components required for a furnace operating in a zero-gravity environment. Such devices are either not needed on Earth or are designed and built specifically for each particular application.

From the information gathered in this survey, it appears that most general purpose commercial furnaces or combinations thereof will be too large, too heavy, and will require too much power to be directly usable for space applications.

The use of multi-foil insulation offers a promising prospect for incorporation into a high temperature space furnace design.

The commercial furnace survey information reinforced the conclusion that a versatile multi-purpose furnace concept would be difficult to achieve. At minimum, if space and power is available, two (2) furnaces; a general purpose and special purpose furnace would be a practical compromise for a space manufacturing facility. This intermediate design position can result in flight hardware on a shorter time scale, with cost effectiveness, and with a higher confidence level for success than can be postulated for a multi-purpose facility. However, some loss in design flexibility is realized.

TABLE 2,1

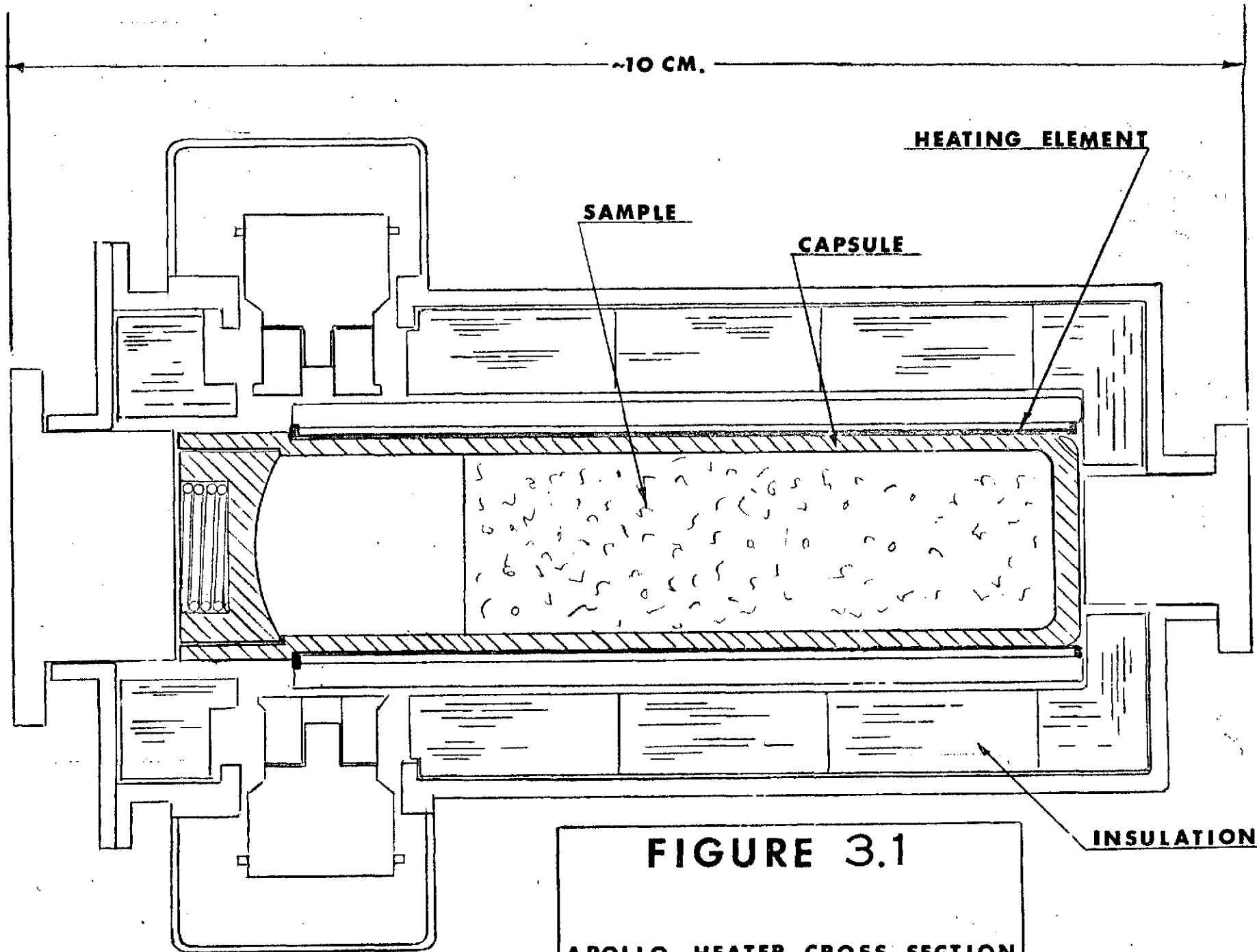
SOME CHARACTERISTICS OF COMMERCIALY AVAILABLE  
GENERAL PURPOSE HIGH TEMPERATURE FURNACES

MANUFACTURER	WORK VOLUME DIMENSIONS	TOTAL VOLUME	POWER REQUIRED FOR MAX. TEMP.	ENVIRONMENT/ SPECIAL FEATURES
Lindberg (Multi-Vac)	10 cm. d x 18 cm. h	.091 m <sup>3</sup>	14KW (2200°C)	Vacuum
Centorr (Model 15-4 X 8-30)	10 cm. d x 20 cm. h	2.94 m <sup>3</sup>	52KVA (3000°C)	Vacuum
High Vacuum Equipment Corp. (Model C-2500-48V)	11.5 cm. d x 20 cm. h	.15 m <sup>3</sup>	37KVA (2500°C)	Vacuum
Edwards High Vacuum (Model RH1200)	5 cm. d x 10 cm. h	.037 m <sup>3</sup>	4KVA (1700°C)	Vacuum-Tungsten Mesh Elements
Vacuum Industries (Minivac)	5.75 cm. d x 10 cm. h	0.8 m <sup>3</sup>	7KVA (2200°C)	Vacuum
Stokes	147 cm. <sup>3</sup>	-	25KW (2300°C)	Vacuum
Thermo Electron (Model 1100-3)	15 cm. d x 15 cm. h	-	3.6KVA (2200°C)	Vacuum-Multi-foil Insulation
Brew (Model 300-MC)	6.5 cm. d x 12.5 cm. h	-	30KVA (3000°C)	Vacuum-Tungsten Mesh Elements
(Model 424B)	11.75 cm. d x 18 cm. h	-	----- (2500°C)	Vacuum-Tungsten Mesh Elements
(Model 520)	10 cm. d x 18 cm. h	-	----- (3000°C)	Argon-Graphite Elements
Leybold-Heraeus	10 cm. d x 10 cm. h	-	5KVA (2400°C)	Vacuum-Multi-foil Insulation
Kanthal (Super 33)	15 cm. x 15 cm. x 15 cm.	-	----- (1800°C)	Oxidizing-MoSi <sub>2</sub> Elements Fibrous Zirconia Insulation
Metals Research Limited (VO 46)	10 cm. d x 10 cm. h	-	6KW (1800°C)	Oxidizing-Alumina Sheated Molybdenum Elements

### 3.0 EARLIER SPACE FURNACE DESIGNS

The Apollo furnace discussed in the previous section, was designed and manufactured by NASA-MSFC. A cross section view of the device is presented in Figure 3.1. The device is simple, compact, safe, and requires low power ( $\sim 34$  watts average). The limitations of the furnace were previously discussed. A further developmental furnace concept has been proposed by Westinghouse Electric Corp. This device shown in Figure 3.2, is a three-chamber furnace capable of temperature operation from 0 to 1000°C consuming less than 100 watts of power. This design has advantages over the earlier MSFC model of slightly increased experiment capacity, versatility and temperature range. Both of these furnaces were designed under rather stringent requirements of power, safety and weight. These requirements are generally dictated by spacecraft and mission constraints. Additionally, both of these furnaces are experimental in nature. In other words, the successful operation of such furnaces is, in itself, an important experiment.

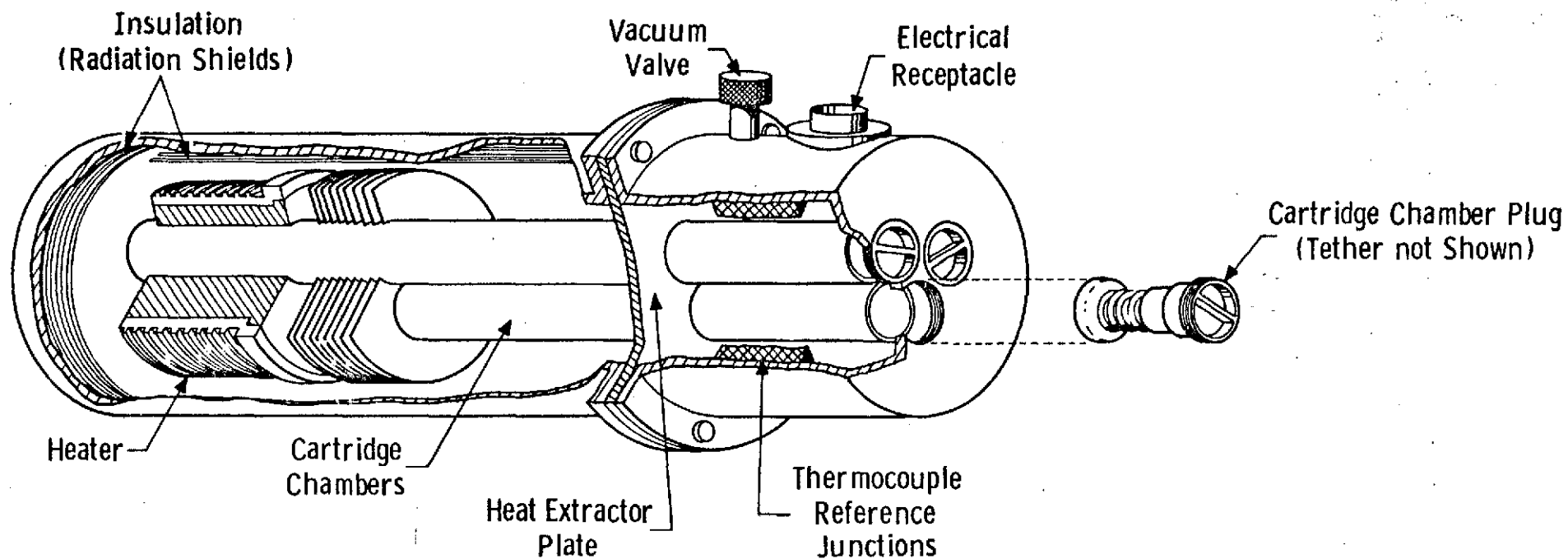
All of the constraints mentioned above have controlled the design of the subject furnaces. They are not capable of general purpose high temperature experimentation and lack the essential ingredient of any good experimental tool; versatility. The results of many of the experiments envisioned cannot be accurately predicted and it is to be expected that extensive real-time modification of experimental techniques will be necessary. Neither of the two operational space furnaces have this built-in capability. The upcoming space missions should allow many of these constraints to be reduced. It will, therefore, be possible to design a furnace which will have the required capabilities.



**FIGURE 3.1**

**APOLLO HEATER CROSS SECTION**





**Fig. 3.2**—Concept for multipurpose electric furnace

## 4.0 THE HIGH TEMPERATURE RADIATING FURNACE DESIGN CONCEPT

### 4.1 Design Concept

The design presented here is an entirely new and unique concept for a zero-gravity furnace. It is capable of operation in a wide range of temperatures and atmospheric conditions. It can hold a sample of up to approximately 10 to 20 kilograms mass (depending upon the sample material), and allows both direct solid manipulation (stirrer) or electro-magnetic (containerless) manipulation. It can be tailored to perform experiments in transient or steady state temperature conditions and gradients. The entire configuration is modularized in order to allow rapid and simple parts and sample replacement and replacement of special purpose components. Future changes dictated by experimental considerations can be accommodated with a minimum of furnace redesign and requalification.

The furnace is designed to minimize heat loss and sample heat-up time. Experimental versatility was the primary design consideration for this multi-purpose furnace.

### 4.2 Furnace Design

The conceptual design is shown in Figure 4.1. The furnace consists of: (1) An outer housing, which functions as a pressure hull and cooling jacket, open at one end to allow internal access; (2) a high-efficiency thermal insulation packaged in a structural pressure housing; (3) a high-temperature three phase heater element; (4) a refractory metal splash shroud mounted to the door which physically isolates the sample (5) a sample manipulation sting; (6) an electro-magnetic or electro-static sample positioning array; and, (7) a quartz window for radiation pyrometry. The outer housing, door/shroud assembly, heater units and positioners form a modularized assembly. Each module can be replaced either for purposes of experimental versatility or refurbishment.

A summary of the furnace design criteria and selected concept details is presented in Table 4.1. A parametric trade-off evaluation, to be discussed later, provided a basis for this design. The furnace design concept selected has high temperature capability, adequate work volume, and can operate in oxidizing atmospheres if a ceramic pressure tight shroud is substituted for the metal shroud.

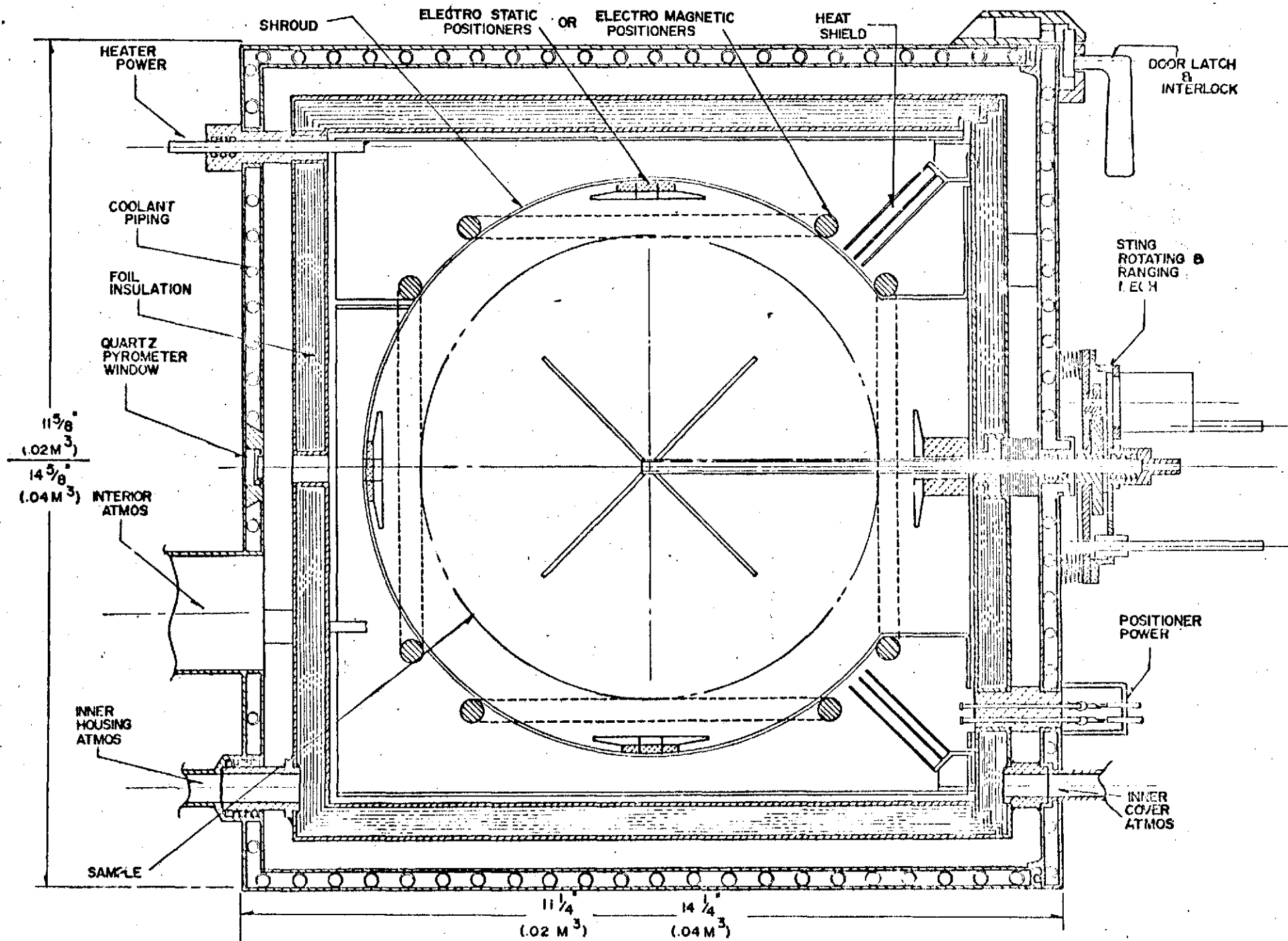


FIGURE 4.1  
HIGH TEMPERATURE FURNACE CONCEPTUAL DESIGN

**TABLE 4.1**  
**DESIGN CRITERIA AND DESIGN CONCEPT**

	<u>OPTION #1</u>	<u>OPTION #2</u>
<b>A. <u>Criteria</u></b>		
Maximum Continuous Temperature	1600°C	2300°C
Maximum Furnace Size	.02 m <sup>3</sup> (.71 ft <sup>3</sup> )	.04 m <sup>3</sup> (1.42 ft <sup>3</sup> )
Maximum Furnace Weight	10 Kg (22 lb)	30 Kg (66 lb)
Maximum Power Input	6 Kw	9 Kw
Sustained Power Input	.3-2 Kw	.3-4 Kw
<b>B. <u>Concept</u></b>		
Maximum Continuous Temperature	2300°C	
Furnace Size	.0195 m <sup>3</sup> (.688 ft <sup>3</sup> )	
Furnace Weight	29 Kg (64 lb)	
Maximum Power Input	6 Kw	
Sustained Power Input	4.5 Kw	

The oxidizing atmosphere is fully contained within the shroud. An inert gas fill of the remainder of the chamber would then be used to minimize shroud leakage and eliminate pressure stresses.

It should be noted that the conceptual design presented is considered to be the best of many options from the standpoint of meeting or approaching the initial specifications. Options to be discussed later in the report allow lower weight, as well as larger sample sizes, and lower power losses. An ordering of the priorities of power, volume, weight, and experimental versatility is necessary before the furnace design can be finalized.

## 5.0 DESIGN CONSIDERATIONS

### 5.1 Experimental Requirements

An investigation was performed to establish the broad requirements for high temperature radiation furnaces for use in orbital laboratories and workshops. Literature on materials sciences and manufacturing in space prepared by NASA personnel and contractors was reviewed. Additional data were obtained from NASA-MSFC personnel.

Twenty-five experiments in seven major categories were identified (Ref. 2-6). These are listed in Table 5.1. Also in the table are outlined the furnace requirements (where known) for each experiment. Factors considered include maximum temperature, required atmosphere, heating or cooling rate, temperature gradient and required manipulative ability. A review of Table 5.1 clearly indicates that few of the proposed experiments require temperatures in excess of 1600°C. The only exceptions identified were titanium-base metal composites, zirconium base nuclear fuel or control material, and certain single crystal and whisker manufacturing processes involving sapphire or similar ceramics. Even in these cases, appropriate data could probably be obtained with a 1600°C furnace by substituting lower melting materials in the experiments.

Another point worth noting is the absence of an identified requirement for oxidizing or reducing atmospheres at temperatures above 1600°C. Limiting the 2300°C furnace to vacuum or inert atmosphere operation will simplify the design; consequently, this approach is recommended for future consideration.

Controlled cooling rates, either slow or rapid, will be required in numerous experiments as will controlled temperature gradients. Furnace design must take this into account. Control of heating rates does not appear to be a critical factor.

TABLE 5.1

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT		FURNACE REQUIREMENTS				
Objective	Approach	Temp. °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>GLASSES</u>						
Laser glasses w/high damage thresholds	Avoid heterogeneous nucleation-containerless melting	<< 1600	Any or all	Controlled cooling	No	Containerless melting
Striation free glasses (Low Index-High Abbe Number glasses)	Avoid convective currents	<< 1600	Vac., H <sub>2</sub> , air, etc.	Controlled cooling	No	None
New glass products, Christiansen filters, passive Q-Switches, Phototropic glasses	New formulations made possible by container- less melting and/or supercooled solidifi- cation and/or particle dispersion	<< 1600	Vac., inert air	Controlled cooling	No	Containerless melting in some cases
Improved lenses and mirrors		<< 1600	Vac., inert air	Controlled cooling	No	Use electric field to rotate and/or shape blanks
Perfect proof mass for advanced acceler- ometers and g-sensors	Free formed blanks and shapes	-	-	-	-	-

TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT		FURNACE REQUIREMENTS				
Objective	Approach	Temp °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>METAL COMPOSITES - ALLOYS AND INTERMETALLIC COMPOUNDS</u>						
Solid Lubricants e.g., Cu-Pb > 30% Pb	Melting or coarse dispersion	<< 1600	Vac., inert	May require rapid cooling	No	Homogenization or emulsifica- tion
New electronic material, e.g., Fe-Pb	Immiscible metal com- ponents, zero-g required	< 1600	Vac., inert	Controlled cooling	Yes	Directional solidification
Blends of low melting metals with high melting particles		<< 1600	Vac., inert, oxidizing or reducing	May require rapid cooling	No	Homogenization
Addition of particles and fibers to low melting metals		<< 1600	Vac., inert, oxidizing or reducing	May require rapid cooling	Yes	Homogenization



TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT		FURNACE REQUIREMENTS				
Objective	Approach	Temp °C	Atmosphere	Heating Cooling Rate	Gradient	Manipulative Ability
<u>METAL COMPOSITES - PARTICLE DISPERSED METAL COMPOSITES</u>						
Fine grained anisotropic castings	Dispersion of seed particles in melt	< 1600	Any or all	Controlled cooling	Yes	None except that needed to maintain gradient
Dispersion strengthened alloys from melt (superalloy and titanium)	Uniform dispersion of oxide particles in melt	< 1600 Superalloy < 2300 Titanium	Vac., inert	Not critical	None	Mold? Homogenization force
Optimized dispersion of fuel or poison in reactor fuel elements or control rods	Dispersion of fuel particles (or poison) in melts	< 2300	Vac., inert	Controlled cooling (?)	None	Homogenization force
Optimized structure of cemented carbide compacts (iron, nickel, cobalt cementing metals)	Coated carbide particles melted	< 1600	Vac., inert reducing	Controlled cooling	None	None

TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT		FURNACE REQUIREMENTS				
Objective	Approach	Temp °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>METAL COMPOSITES - WHISKER DISPERSED METAL COMPOSITES</u>						
Light metal components with high strength/weight and stiffness/weight ratios, e.g., $Al_2O_3$ in Al	Uniform dispersion in melt	$\ll 1600$	Any	Not critical	No	Electromagnetic or mechanical vibration
High temperature components with better creep and oxidation properties, e.g., SiC in Ni or cobalt alloys	Uniform dispersion in melt	$< 1600$	Any	Not critical	No	Electromagnetic or mechanical vibration

TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT		FURNACE REQUIREMENTS				
Objective	Approach	Temp °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>METAL COMPOSITES - DENSITY CONTROLLED METALLIC COMPOSITES</u>						
Lightweight components with high compressive strength/weight for deep sea use	Foamed metals with uniform controlled size bubble structure	< 1600	Vacuum	Not critical	No	Gas injection mechanism
Pressure stiffened ingots for directional expansion in molds on earth						
New category of structural components with superior comb. of strength, density	Foaming of melts containing dispersed fibers and/or whiskers	< 1600	Vacuum	Not critical	No	Vibration for homogenization plus gas injection
Variable density components, e.g., stress turbine blades	Centrifugal distribution of bubble dispersion	< 1600	Vacuum	Not critical	No	Gas injection mechanism plus cyclic pulse system to spin melt

TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENTS		FURNACE REQUIREMENTS				
Objective	Approach	Temp °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>CRYSTALS</u>						
Improved single crystals for semiconductor, optical, piezoelectric, ferroelectric applications	Contact free growth, uniform composition and dopant dispersion	< 1600	Vac., inert	Slow controlled cooling in solution grown case	Yes, in melt grown case or in place of controlled cooling in solution grown cases	Containerless melting, homogenization method (vibration) in some cases
Continuous pulling of perfect single crystals and fibers	Contact free melting	< 2300	Vac., inert air			Containerless melting
Crystal whiskers of high strength and high aspect ratio	Vapor grown crystals (?)					

TABLE 5.1 (Continued)

REQUIREMENTS FOR HIGH TEMPERATURE RADIATION FURNACES IN SPACE

EXPERIMENT			FURNACE REQUIREMENTS			
Objective	Approach	Temp °C	Atmosphere	Heating or Cooling Rate	Gradient	Manipulative Ability
<u>CONFIGURATIONS</u>						
Ingots w/oriented structure for terrestrial fabrication	Directionally solidified eutectic	<< 1600	Vac., inert	Controlled cooling	Yes	Only that associated with directional solidification
Perfect spheres, spheroids, other symmetrical shapes	Shaping in liquid state by surface tension alone or with inertia or electric field forces	< 1600	Any or all	No	No	System for application of shape forces
Other geometric shapes, e.g., single crystal ribbon	Drawing of membranes and filaments from liquid	< 2300	Any or all	Controlled cooling	Yes	System for application of shape force

Several types of manipulation, either singly or in combination, will be needed to perform the various experiments. These include:

1. A means to maintain the position of materials for containerless melting.
2. A means to apply shaping forces to molten materials.
3. A means for mixing or homogenizing liquids or liquid-solid mixtures.
4. A means for casting molten metals into molds.
5. A gas injection or foaming method.

All these manipulations will be required at 1600°C, but only the first three are needed at 2300°C.

## 5.2 Design Requirements

Five design goals presented in Table 5.2 were specified by MSFC at the inception of this program. It is stressed that future changes in these criteria can have a significant effect upon the ultimate feasibility of the proposed furnace design concept.

## 5.3 Versatility Considerations

As stated above, the key consideration of the design concept is versatility. To attain a high level of versatility, the furnace will be of modular form. This consideration allows a variety of experiments to be performed and on-site replacement of defective parts. As furnace experience develops in orbit, additional experiments will be envisioned. In-orbit parts replacement or substitution will minimize down time and overall costs. For example, a 1600°C experiment in vacuum can be quickly followed by a 1300°C experiment in oxygen by simply replacing the metal shroud with its sealed ceramic counterpart. Experiment turn-around time for this furnace can be reasonably rapid.

## 5.4 Material Considerations

Materials used within the insulation assembly will be subject to substantially the same temperatures as the sample. All internal furnace materials must therefore be suitable for service at 1600°C and/or 2300°C. The components subject to these temperatures are as follows:

TABLE 5.2  
FURNACE DESIGN GOALS

	<u>OPTION #1</u>	<u>OPTION #2</u>
Maximum Continuous Temperature	1600°C (2900°F)	2300°C (4200°F)
Maximum Furnace Size	.02 m <sup>3</sup> (.71 ft <sup>3</sup> )	.04 m <sup>3</sup> (1.42 ft <sup>3</sup> )
Maximum Furnace Weight	10 Kg (22 lb)	30 Kg (66 lb)
Maximum Power	6 Kw	9 Kw
Sustained Power	.3-2 Kw	.3-4 Kw

- Sample
- Support Sting
- Shroud
- Heater Electrodes
- Insulation Housing
- Insulation
- Positioner Devices

It is necessary that these components be fabricated from materials which can withstand such temperatures. Molybdenum alloys are suitable for 1600°C operation, while tantalum or tungsten alloys are required for 2300°C operation. Non-metallic parts may be made from various metal oxide-ceramics.

Refractory alloys are not suitable for direct contact with oxidizing atmospheres above approximately 300-400°C. Cobalt and nickel alloys are suitable to 1000-1100°C. Below 1300°C, it is possible to use noble metal alloys (Pt, Rh, Ir) for the critical parts. However, the cost increment for such parts may be prohibitive. In addition, all noble metal alloys exhibit substantial creep strain at low levels of stress above 1000°C. The only suitable method for component oxidation protection above approximately 1300-1400°C is total isolation from the oxidant. A ceramic-oxide shroud will serve as both a sample holder and test atmosphere isolater. This shroud must be pressure tight and possess reasonable strength and thermal conductivity up to the test temperature.

The cooling jacket is subject to a maximum 41°C (105°F) temperature and can therefore be made of light aluminum or magnesium alloys.

Summaries of material properties and recommended materials are presented in Appendices A and B respectively.

## 5.5 Structural Considerations

The furnace will be subjected to a number of structural loads. It must be capable of maintaining its structural integrity during launch acceleration and vibration. During operation, it must be capable of supporting pressure gradients in several directions. Replacement parts must not be degraded by handling, removal or installation loads.



It was assumed for this study that suitable means are available to support the furnace components during launch. Support of fragile components by removable inserts should prove satisfactory and straightforward.

Components subject to pressure stress during operation include the cooling jacket, when the internal furnace volume is evacuated, and the insulation housing when the internal furnace volume is pressurized. For safety considerations, the highest internal furnace pressure was assumed to be the same as the cabin pressure (although chemically different). Standard cabin pressure for space vehicles can be up to 1 atmosphere.

There are discussions in process to agree on a uniform cabin pressure for U.S.A. and U.S.S.R. spacecraft. Present indications are that the value may be in the range of .7 to 1.0 Atm. Cabin pressures in the range should not substantially alter the furnace design. However, it can influence some experimental details. A structural analysis of the expected pressure loads and critical pressures is presented in Appendix C.

## 5.6 Thermal Considerations

### 5.6.1 Spacecraft Thermal Considerations

The thermal load of the furnace on both the space vehicle power supply and heat removal systems is one of the most critical integration factors to be considered. The transient and concentrated nature of this power must not tax these systems. The Initial Space Station (ISS) experiment power is estimated at 4.8 Kw (Ref. 5-9). The Growth Space Station (GSS) power is estimated at 12.1 Kw (Ref. 7-9). It is clear that a 6 Kw furnace heat-up in the ISS is only possible if some type of energy storage system is available. Storage batteries could be used for this purpose. For example, 150 to 200 lbs. of nickel-cadmium batteries can provide 3 Kw start-up power for approximately 30 minutes. Heat-up rates below 4.8 Kw are possible, but will require substantial times. Minimum energy consumption is achieved by minimizing heat-up time. Additionally, longer heat-up times may produce priority conflicts with other on-board experiments. The GSS available power appears adequate for furnace operation but may still generate some experiment conflicts.

### 5.6.2 Furnace Thermal Considerations

The initial requirements designated investigation of two maximum temperature regimes for furnace operation of 1600°C and 2300°C. Unshielded furnaces operating at these temperatures would radiate approximately 100 and 300 Kw of thermal energy respectively for the sizes under consideration. Heat rejection rates are reduced to manageable levels by proper design of thermal insulation and minimization of parasitic heat paths. The imposed limitations on power usage are chiefly controlled by the power availability (primarily from the vehicle primary system) and heat removal systems available on the spacecraft. For the purpose of this study, the allowable maximum and sustained heat rates were fixed at 6 Kw and 2 Kw respectively for the 1600°C furnace and; 9 Kw and 4 Kw respectively for the 2300°C furnace. Heat losses are minimized and work volume is maximized by the use of vacuum-foil superinsulation. The thermal resistivity of the insulation is so high that only 2% to 4% of the system heat losses are attributable to the insulation assembly. The remaining losses occur through the insulation walls including joint areas and equipment feed-through points. This insulation has several potential disadvantages. It is inherently weak structurally and requires a vacuum environment ( $< 10^{-5}$  torr) for design operation. A rigid housing is provided to locate and support the insulation during non zero-gravity conditions and when the furnace is pressurized with a reducing, oxidizing, or inert gas environment. Heat transferred through the housing, insulation and associated equipment is collected by the cooling jacket and subsequently transferred to the spacecraft heat removal system.

Key thermal considerations are presented in Table 5.3. Items 1, 2 and 3 are of primary importance at this stage of concept development.

In order to evaluate the thermal response of the furnace, a digital heat transfer computer program was developed to mathematically model the thermal response. This model was used to compute component temperatures and heat loads for both steady state and transient cases. The effects of housing materials and thickness, test gases, furnace size, sample size, and input power on the thermal response of the system were considered.

TABLE 5.3

FURNACE THERMAL CONSIDERATIONS

1. Reduction of power requirements to within specified maximums.
2. Determination and minimization of component temperatures (other than sample).
3. Prevention of hazardous external "Hot Spots" on the furnace.
4. Minimizing sample heat-up time.
5. Production of uniform, reproducible test temperatures.

A detailed discussion of the thermal model and analysis methods used and a presentation of the thermal response results are given in Appendix D.

#### 5.7 Volume Considerations

The allowable volume for the two furnace options initially indicated (Table 5.2) was used to analyze the conceptual designs. A cylindrical form with the diameter approximately equal to the length was chosen to maximize work volume and minimize heat losses.

#### 5.8 Weight Considerations

The furnace concept presented in Section 4 is considered to be the best option to achieve the design goals. A number of variations are available by which versatility of the furnace can be increased. A summary of all pertinent design options, including weight, is presented in Tables 5.5 and 5.6.

The weight estimates are based upon pessimistic structural assumptions and should, therefore, represent maximum weight conditions. Option #1 is the lightest version at 12 Kg. It is capable of operation to 1600°C in vacuum, inert, or reducing atmospheres to a maximum test pressure of approximately .3 Atm. Increased furnace functions are obtained at the expense of increased weight, as indicated by the subsequent entries in the table.

Oxidizing atmosphere operation in the 1600°C-2300°C range is possible only if all the refractory metal components are isolated from the oxidizer. These materials oxidize rapidly at such temperatures. Under conditions of sufficient pressure, combustion can occur. Commercial oxygen furnaces generally protect such components with oxide ceramic sheathing. It is not practical to sheathe all oxidation-prone components in the furnace. The weight and heat penalties would be too great. A more practical approach is to sheathe the sample in its oxidizing atmosphere. This can be accomplished as shown in Figure 5.1. The shroud is made from an oxide-ceramic suitable for the specific experiment. A hollow ceramic sting is used to transport the oxidizing gas to the test site. Leakage is minimized by the use of one or more vacuum traps. The remaining portions of the furnace are pressurized with inert gas to the same or slightly higher pressure. This technique should eliminate any leakage. If leakage remains a problem, it is also possible to use a reducing gas rather than an inert gas. This gas will combine (by combustion)

TABLE 5.5

FURNACE DESIGN CHARACTERISTICS

Design Option #	Design Details	Estimated Volume (m <sup>3</sup> )	Estimated Mass (Kg)	Insulation Housing Construction Material and Thickness (mm)
1	Small furnace; vacuum, inert, reducing atmosphere at <.3 Atm to 1600°C	.0195	< 12	Molybdenum, .5mm
2	Small furnace; vacuum, inert, reducing atmosphere at <1.0 Atm to 1600°C	.0195	< 16	Molybdenum, 1.27mm
3	Small furnace; vacuum, inert, reducing, oxidizing atmosphere at <.3 Atm to 1600°C	.0195	< 14*	Molybdenum, .5mm with ceramic shroud
4	Small furnace; vacuum, inert, reducing, oxidizing atmosphere at < 1. Atm to 1600°C	.0195	< 18*	Molybdenum, 1.27mm with ceramic shroud
5	Small furnace; vacuum, inert, atmosphere at < .2 Atm to 2300°C	.0195	< 17	Tantalum, .5mm
6	Small furnace; vacuum, inert, reducing atmosphere at < .03 to 2300°C	.0195	< 19	Tungsten, .5mm
7	Small furnace; vacuum, inert, atmosphere at <.3 Atm to 2300°C	.0195	< 25	Tantalum, 1.27mm
8	Small furnace; vacuum, inert, reducing atmosphere at <.3 Atm to 2300°C	.0195	< 27	Tungsten, 1.27mm

\*All furnaces utilizing an oxidizing atmosphere have a ceramic pressure tight shroud. This increases the weight by approximately 2 Kg.

TABLE 5.5

## FURNACE DESIGN CHARACTERISTICS

Design Option #	Design Details	Estimated Volume (m <sup>3</sup> )	Estimated Mass (Kg)	Insulation Housing Construction Material and Thickness (mm)
9	Small furnace; vacuum, inert, reducing and oxidizing atmosphere at < .03 Atm to 2300°C	.0195	< 21	*Tantalum, .5mm with ceramic shroud
10	Small furnace; vacuum, inert, reducing and oxidizing atmosphere at < .3 Atm to 2300°C	.0195	< 29	*Tantalum, 1.27mm with ceramic shroud
11	Large furnace; vacuum, inert, reducing atmosphere at < .03 Atm to 1600°C	.039	< 18	Molybdenum, .5mm
12	Large furnace; vacuum, inert, reducing atmosphere at < .3 Atm to 1600°C	.039	< 24	Molybdenum, 1.27mm
13	Large furnace; vacuum, inert, reducing, oxidizing atmosphere at < .03 Atm to 1600°C	.039	< 21	*Molybdenum, .5mm with ceramic shroud
14	Large furnace; vacuum, inert, reducing, oxidizing atmosphere at < .3 Atm to 1600°C	.039	< 27	*Molybdenum, 1.27mm with ceramic shroud
15	Large furnace; vacuum, inert, atmosphere at < .03 Atm to 2300°C	.039	< 25	Tantalum, .5mm
16	Large furnace; vacuum, inert, reducing atmosphere at < .03 Atm to 2300°C	.039	< 28	Tungsten, .5mm

\*All furnaces utilizing an oxidizing atmosphere have a ceramic pressure tight shroud. This increases the weight by approximately 2 Kg.

TABLE 5.5

FURNACE DESIGN CHARACTERISTICS

Design Option #	Design Details	Estimate Volume (m <sup>3</sup> )	Estimated Mass (Kg)	Insulation Housing Construction Material and Thickness (mm)
17	Large furnace; vacuum, inert, atmosphere at < .3 Atm to 2300°C	.039	< 37	Tantalum, 1.27mm
18	Large furnace; vacuum, inert, reducing atmosphere at < .3 Atm to 2300°C	.039	< 41	Tungsten, 1.27mm
19	Large furnace; vacuum, inert, reducing and oxidizing atmosphere at < .03 Atm to 2300°C	.039	< 32	*Tantalum, .5mm with ceramic shroud
20	Large furnace; vacuum, inert, reducing and oxidizing atmosphere at < .3 Atm to 2300°C	.039	< 44	*Tantalum, 1.27mm with ceramic shroud

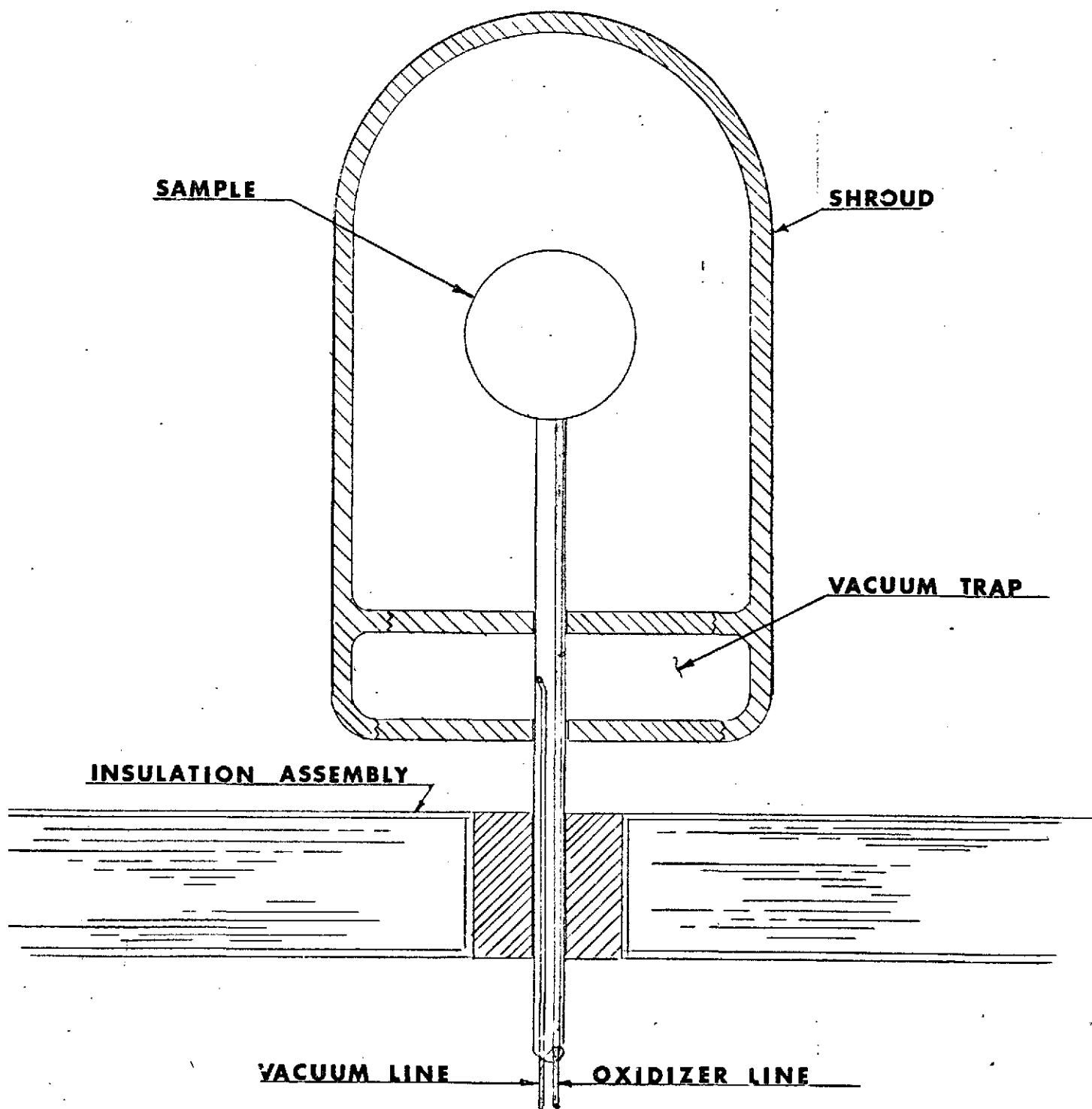
\*All furnaces utilizing an oxidizing atmosphere have a ceramic pressure tight shroud. This increases the weight by approximately 2 Kg.

TABLE 5.6  
WEIGHT BREAKDOWN - LIGHTEST WEIGHT FURNACE

Small Furnace, .5mm Housing, Molybdenum, for 1600°C Operation

<u>COMPONENT</u>	<u>ESTIMATE WEIGHT</u>
Cooling Jacket . . . . .	2.7 Kg (6 lb)
Positioner Coils, Sting . . . . .	1.4 Kg (3 lb)
Auxiliary Drive Gear . . . . .	.45 Kg (1 lb)
Insulation Housing (.5mm). . . . .	3.1 Kg (6.8 lb)
Insulation (.01mm/Layer, 80 Layers) . . . .	2.4 Kg (5.3 lb)
Heater . . . . .	2.7 Kg (6 lb)
<hr/>	
TOTAL	12.8 Kg (28 lb)
<hr/>	





**FIGURE 5.1**

**CERAMIC SHROUD WITH VACUUM TRAP**

with any leaking oxidant. This method is feasible and safe and has been used in industrial furnaces, (Ref. 10), as long as the leak rates are low enough to prevent the accumulation of an explosive mixture.

It is apparent from this study that it will be difficult to meet the 10 Kg and 30 Kg weight specifications under all possible test conditions. The ultimate choice of furnace options depends upon the relative importance of the initial furnace requirements and the desired experimental capability. A trade-off of weight versus experimental capability is needed.

#### 5.9 Integration Considerations

The high temperature radiating furnace must be capable of integration with a space vehicle. The furnace can be located in a General Purpose Laboratory (GPL) module as part of the Mechanical Sciences Laboratory Facility.

The peak power load (6-9 Kw) and the sustained power load (3-5 Kw) of the furnace can represent a substantial percentage of available power (e.g. 12.1 Kw for the GSS). Furnace experiment planning must be coordinated with other experiments so that conflicts are minimized.

The space vehicle heat removal subsystem must be capable of removing the 3-5 Kw sustained power losses of the furnace.

Electrical interlocks to prevent furnace operation during periods of excess power consumption or insufficient space vehicle heat removal capability will be necessary.

The experiment control logic must be capable of controlling the input power level and coolant flow rate. It should also be capable of automatic sequencing procedures to reduce or eliminate the necessity of human intervention during experiments.

Suitable consumables must be on-board for experimental use. Inert, oxidizing, or reducing gases will be necessary for various experiments. It is also possible that such gases would be incorporated with each experiment sample, as required.

#### 5.10 Safety Considerations

The furnace will require a man-rating program to establish safety and operations requirements. The following safety-

related factors must be observed:

- 5.10.1 The internal furnace pressure must never be equal to or greater than the cabin pressure. This will prevent the escape of hot or toxic materials from the furnace.
- 5.10.2 Interlocks to prevent the opening of the furnace during an experiment and until the internal pressure and temperature are within safe limits.
- 5.10.3 The use of reducing (i.e. hydrogen) test environments may constitute a definite safety hazard in case of leakage. Sensors to measure leakage and cause evacuation of the furnace if leak rates exceed specified limits are recommended.
- 5.10.4 Extended use of the furnace will lead to a gradual degradation of the insulation and increase heat rejection requirements. Safety interlocks will be required on the cooling system to stop operations in the event of an over-temperature situation.
- 5.10.5 An emergency cooling capability must be incorporated into the furnace. This will include a provision to flood the insulation with helium. This will increase the rate of heat transfer and cool the furnace more rapidly. If faster cooling is required, it is possible to flood the entire furnace with an inert cooling gas. In no instance must the cabin gas be used for this purpose. At the planned operating temperatures of 1600°C or 2300°C, the refractory metal parts of the furnace will ignite in the presence of oxygen under sufficient pressure. Such an occurrence could destroy the entire furnace and constitute a serious personnel and vehicle safety hazard in a matter of a few seconds.

## 6.0 CONCLUSIONS AND RECOMMENDATIONS

- 6.1 The high temperature radiation furnace concept presented herein represents a unique approach to the problem of zero-gravity materials processing. Its temperature capability is suitable for most materials of interest; it can operate with vacuum, inert, reducing, or oxidizing environments; and, it can accommodate samples of significant mass and dimension. Most program design goals were achieved.
- 6.2 A modularized design concept was evolved. This design philosophy allows for rapid facility maintenance and ease of component and subsystem replacement. In addition, experiment related functions and equipment can be separated from facility construction, thus the basic structure can be directly integrated into the space vehicle compartment and that portion of the furnace directly associated with the experiment can be made available on an interchangeable basis.
- 6.3 Furnace input power, heat rejection requirements, and integration characteristics are within the capabilities of anticipated space laboratory facilities of the near future.
- 6.4 The analysis of candidate furnace experiments indicates that the majority can be accommodated with a 1600°C furnace.
- 6.5 The smaller volume furnace ( $0.0195 \text{ m}^3$ ) is well suited to the matrix of anticipated experiment requirements. Therefore, it is recommended that future investigations relative to a multi-purpose furnace be limited to concepts of this volume and to a temperature capability of up to 2300°C. Ample work space is available to accomplish all anticipated manufacturing functions.
- 6.6 No contemporary commercial furnaces can meet the major program design and performance goals of power, weight, volume, and versatility. In addition, even with major modifications, none of them can satisfy these objectives. Based upon the work performed under this study, two furnaces may offer significant advantages over a single multi-purpose facility concept and should be considered for further investigation. This compromise design position can result in flight hardware on a shorter time scale, more cost effectively and with a higher confidence level of success than can be postulated for a

single general purpose facility. Each furnace should be limited to a volume of about  $.02 \text{ m}^3$  and have temperature capability to  $2300^\circ\text{C}$ .

- 6.7 Available space vehicle power appears to be the major design restriction relative to furnace facilities. The small furnace ( $.0195 \text{ m}^3$ ) requires approximately 30% less power than the large furnace ( $.039 \text{ m}^3$ ) under similar test conditions.
- 6.8 Several design features of the concept presented herein cannot be specifically quantified at this time, e.g., containerless melting and sample stirring techniques. Included within the presented design are provisions for electro-static and/or electro-magnetic levitation devices and ultrasonic or other advanced techniques for stirring processes.

## 7.0 REFERENCES

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## APPENDIX A

### Material Thermal Properties

Presented below are the material properties used in the Task 500 Parametric Analysis.

Heat Capacity and Thermal Conductivity are standardized in the forms:

$$C_p = A + BT \text{ (}^\circ\text{C)} \text{ cal/gm - }^\circ\text{C}$$

$$K = C + DT \text{ (}^\circ\text{C)} \text{ watts/cm - }^\circ\text{C}$$

TABLE A-1 MATERIAL THERMAL PROPERTIES

Material	A	B	C	D	Total Hemispherical Emissivity
Aluminum	.22	0	$2.04 \times 10^{-3}$	0	.2
Argon	.124	0	$2.02 \times 10^{-4}$	$2.52 \times 10^{-8}$	--
Helium	1.24	0	$5.09 \times 10^{-4}$	$2.18 \times 10^{-6}$	--
Hydrogen	3.3	$4.212 \times 10^{-4}$	$1.97 \times 10^{-3}$	$3.15 \times 10^{-6}$	--
Iron	.157	0	.398	0	.2
Molybdenum	.049	$2.52 \times 10^{-5}$	1.41	$3.89 \times 10^{-4}$	.4
Nitrogen	.156	$2.12 \times 10^{-5}$	$2.57 \times 10^{-4}$	$5 \times 10^{-7}$	--
Oxygen	.242	$2.29 \times 10^{-5}$	$3.46 \times 10^{-4}$	$5.59 \times 10^{-7}$	--
Tantalum	.0335	$9 \times 10^{-6}$	.6	$-1.24 \times 10^{-4}$	.4
Tungsten	.031	$5.85 \times 10^{-6}$	1.268	$1.25 \times 10^{-4}$	.3

## APPENDIX B

### Material Properties

From a materials standpoint the inner furnace components are most critical. These components include the heating elements, the shroud, the sting and the foil insulation package. The selection of materials of construction for these components is constrained by the required operating temperature, mechanical loads, and experimental environment.

The presented furnace allows the flexibility of selecting construction materials for the inner components which are optimal in regard to weight and thermal properties within the constraints of the operating parameters.

For example, experiments may be performed up to 1300°C in oxidizing, reducing, vacuum and inert gas environments using conventional high temperature furnace hardware such as silicon carbide and molybdenum disilicide as heating element, shroud and sting and platinum alloys for the foil insulation package.

For operation between 1300-1600°C high strength platinum or iridium base alloys could be used under the total spectrum of environmental conditions. The final selection of materials for this temperature range would require more detailed analyses, but the option of using the oxidation resistant platinum and iridium alloys can be kept open.

Above 1600°C one is restricted to the refractory metals and their alloys. Molybdenum, tantalum and tungsten alloys are available for these applications. Molybdenum and molybdenum base alloys would be leading candidate materials for 1600-2000°C operation in vacuum, reducing and high purity inert gas environments. The lower density of molybdenum makes it a more desirable choice than tantalum and tungsten alloys.

Above 2000°C the choice rests between tantalum and tungsten alloys. Tantalum alloys possess lower densities and thermal conductivities than tungsten alloys and are more easily fabricated and welded. Tantalum alloys are limited to vacuum and inert gas environments while tungsten alloys can also be used in a hydrogen (reducing) environment.

In order to conduct experiments in an oxidizing atmosphere above 1600°C it will be necessary to isolate the heating elements and the foil insulation from the working material with a totally enclosed oxidation resistant shroud. For 1600-2000°C operation a high purity alumina ( $\text{Al}_2\text{O}_3$ ) shroud would be adequate while above 2000°C, zirconia ( $\text{ZrO}_2$ ) or thoria ( $\text{ThO}_2$ ) would be required.



Table B-1 shows some properties of typical candidate furnace materials while Figure B-1 shows their temperature and environmental limitations.

TABLE B-1

TYPICAL PROPERTIES OF CANDIDATE FURNACE MATERIALS

	Melting Point (°C)	Density (G/cc)	Thermal Conductivity	Ultimate Strength lbs/in <sup>2</sup>
Tungsten Tungsten-25Re	3410°C 3100°C	19.3 g/cc 19.6 g/cc	0.39 cal/cm-sec-°C	1600°C - 40,000 psi 1600°C - 35,000 psi
Tantalum T-111	3000°C 2982°C	16.6 g/cc 16.7 g/cc	0.13 cal/cm-sec-°C	1650°C - 11,000 psi 1650°C - 20,500 psi
Molybdenum Molybdenum-50Re	2630°C 2550°C	10.2 g/cc 13.76 g/cc	0.34 cal/cm-sec-°C	1300°C - 25,000 psi 1600°C - 15,000 psi
Iridium Iridium - 20/40 Rh	2410°C 1982 - 2148°C	22.5 g/cc 14.42-16.44g/cc	0.35 cal/cm-sec-°C	1500°C - 22,600 psi 1300°C - 12-37,000 psi
Platinum Platinum 30 Rh	1769°C 1901°C	21.45 g/cc 16.94 g/cc	0.17 cal/cm-sec-°C	1000°C - 6,000 psi 1300°C - 8,000 psi

FIGURE B-1

TEMPERATURE °C

## APPENDIX C

### Structural Analysis

#### 1.0 Pressure Loading

The furnace is subject to pressure loading during all experiments. During vacuum tests, the cooling jacket must prevent furnace collapse due to cabin pressure. During pressure tests, the insulation housing must not collapse. These two extreme cases are shown schematically in Figure C-1. Two modes of failure are possible, (1) exceeding elastic limits of the material; and, (2) elastic buckling below the elastic limits of the material.

#### 1.1 Elastic Buckling of the Insulation Housing

Elastic buckling can occur when the cylindrical shape of the parts in question becomes a statically unstable configuration under compressive stresses. For Case 1 (furnace evacuated) the cooling jacket is subject to this type of failure. During pressure operation, the outer wall of the insulation housing can buckle. The elastic buckling limits of finite cylinders have been determined empirically to satisfy the following equation:

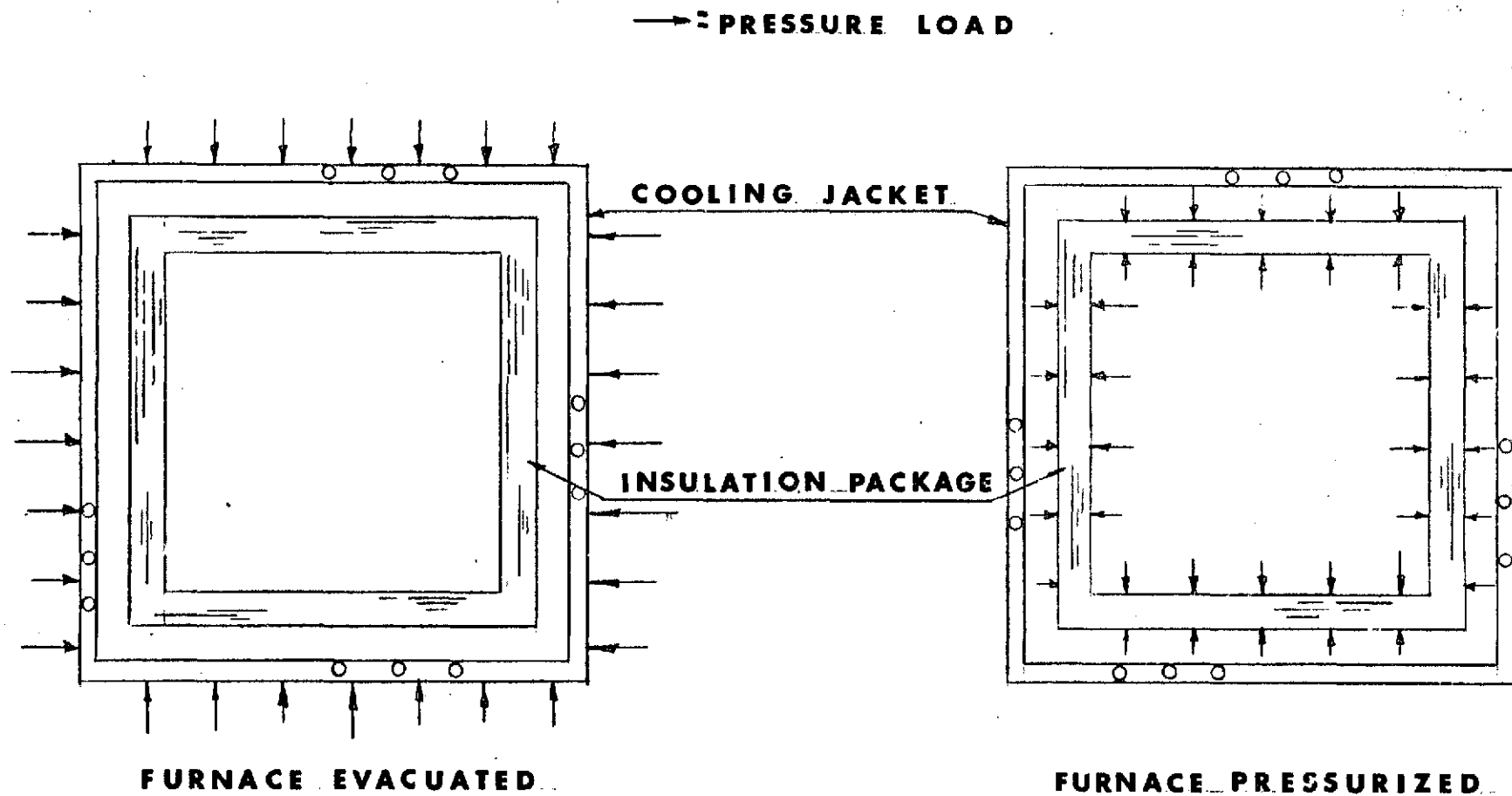
$$P_{crit} = KE \left( \frac{D}{t} \right)^3 \quad (\text{Ref. C-1})$$

where, K, is a unique function of the cylindrical aspect ratio; (L/D) and the finesse ratio (D/t).

The small furnace outer insulation housing wall has the following dimensions:

Thickness	-	.5mm (minimum possible)
Diameter	-	25cm
Length	-	23.5cm
Material	-	1600°C - Molybdenum 2300°C - Tantalum or Tungsten

The value of, K, for the values given above (Ref. C-1) is 65.



**FIGURE C-1**

**FURNACE PRESSURE LOAD SCHEMATIC**

The elastic moduli for the materials and temperatures indicated above are not well defined. Most data presented are for temperatures to 1000-1400°C. However, estimates based on data from Reference C-2 indicate that a value of  $7.03 \times 10^5 \text{ Kg/cm}^2$  ( $1 \times 10^7 \text{ psi}$ ) for molybdenum at 1600°C is not unreasonable. Using this value gives a critical collapse pressure:

$$P_{\text{crit}} = .35 \text{ Atm}$$

The use of two or three stiffener bands, welded around the circumference of the insulation housing should increase this critical value by a factor of 3-5. The .3 Atm limit is therefore based on a safety factor of 3 to 5.

The elastic moduli for tantalum and tungsten at 1600°C and 2300°C were not available. However, data from Reference C-3 indicate that a tantalum elastic modulus of  $8.4 \times 10^5 \text{ Kg/cm}^2$  ( $12 \times 10^6 \text{ psi}$ ) at 1600°C is reasonable. The value for tungsten should be somewhat higher at all temperatures. No elastic data exists for 2300°C operation.

Minimum elastic modulus of  $2 \times 10^6 \text{ psi}$  for both tantalum and tungsten at 2300°C has been assumed. This establishes a critical pressure of approximately 1 Atm for a 1.27mm thickness. Again, it is expected that stiffening bands will increase the critical buckling pressure by a factor of 3 to 5.

The door insulation assembly and the opposite face must also be prevented from collapsing. Stiffening bands should be a suitable solution.

## 1.2 Elastic Buckling of the Cooling Jacket

The cooling jacket can be subject to elastic buckling stresses when the furnace is evacuated. However, the jacket is essentially at room temperature (41°C) and therefore has a modulus of elasticity of approximately  $7.03 \times 10^5 \text{ Kg/cm}^2$  ( $1 \times 10^7 \text{ psi}$ ). The coolant circulation tubing will add significantly to the jacket strength. No collapse problems are anticipated. A 1.27mm wall thickness has been assumed.

## 1.3 Inelastic Buckling or Bursting of the Insulation Housing

For some combinations of materials, shapes, sizes and temperatures, it is possible to exceed the elastic limit

of strain without exceeding the elastic buckling limit. The inner cylindrical wall of the insulation housing will be subject to a positive stress when the furnace is operated with a test atmosphere. The hoop stress in this part is largest, and, in simplified (and conservative) form, can be expressed as,

$$\sigma_{\text{hoop}} = \frac{PR}{t}$$

For the small furnace,  $R \approx 10.9\text{cm}$   
 $t \approx .5\text{mm}$

Therefore,  $\sigma_{\text{hoop}} = 215P \text{ (Atm)}$

If  $P = 1 \text{ Atm}$ ,  $\sigma_{\text{hoop}} = 215 \text{ Kg/cm}^2 \text{ (3060 psi)}$

This is below the yield strength of the materials involved in all cases. The outer wall of the housing will be subject to a compressive stress of slightly higher magnitude, but should still be sufficient.

## 2.0 Structural Analysis Results

- 2.1 The .5 mm molybdenum insulation housing wall thickness appears adequate for the small furnace operating at 1600°C or less with a maximum test pressure of .3 Atm.
- 2.2 Higher temperature (2300°C) operation will require a tantalum or tungsten insulation housing at approximately 1.27mm thickness to be suitable for 1 Atm operation.
- 2.3 The structural data used for this analysis are not sufficiently accurate to allow insulation housing thickness and consequentially weight values to be fixed at this time.

The values generated herein are preliminary values which must be substantiated, possibly with new test data at a later date.

## 3.0 References

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## APPENDIX D

### Thermal Analysis

#### 1.0 Method

The desired performance of the furnace is achieved when heat losses are minimized, heat-up time is minimized, and experimental versatility is maximized. A transient and steady state thermal analysis of the furnace was performed and the thermal response characteristics were determined.

The thermal analysis was conducted by using a thermal model that was based upon a digital finite-difference solution technique. This method approximates the microscopic nature of heat transfer and heat storage with macroscopic mathematical manipulations. The second order differential equations of heat transfer are approximated by algebraic expressions. An iterative technique is used to solve and resolve these approximate equations until the result is very close to the exact solution. The solution of even a simple problem by this technique requires several thousand calculations. A more complex problem may require several million calculations. These equations are therefore solved by computers. The specific program used for this study is entitled the "Thermal Analyzer Program #4" (TAP 4). The original program was written about nine years ago by Atomics International to solve reactor cooling problems. TAP 4 and variations have been in extensive use ever since. This computer code has been used successfully on such aerospace programs as SNAP 19, SNAP 29, and New Moons. Correlations of computer results with test data have been consistently good. The inherent calculational error of the program has been demonstrated many times to be in the 1% to 3% range. It is programmed for use on the Univac 1108 digital computer which was used for this study as well as other computer types used for engineering analyses.

#### 2.0 Thermal Modeling Technique

All furnace components involved in heat transfer are included in the thermal model. Each component is divided into a number of small elements. The internal portions of each element are assumed to be at a uniform temperature. Hence, all temperature-dependent properties are also uniform within the element. A zero-dimensional point, called a "node" is positioned at the centroid of the element. Ascribed to each node are the mass and thermal energy capacity equivalent to the real element it represents. The nodes are connected to each other by conductance paths. Each path has a thermal resistance associated with it. This resistance is determined by the cross sectional area, material conductivity, and distance between centroids of the



elements. These connectors are necessary only where a real conductive heat transfer path exists. Radiative heat transfer is handled in a similar manner. The resulting one, two, or three dimensional array of nodes and connectors is called a mesh. The mesh has an electrical analog wherein the nodes represent capacitors, the connectors represents resistances, heat transfer is equivalent to current, temperature is equivalent to voltage, and thermal energy is equivalent to electric energy.

A typical heat transfer problem is shown with its thermal analog and electrical analog in Figure D-1.

The principal task of the analyst is to devise the thermal analog such as to minimize its complexity while maintaining its thermal accuracy.

In general, a greater node density (i.e. smaller element size) is recommended in areas where high temperature gradients and high heat flux values are expected to exist simultaneously. Preliminary models are often programmed in order to optimize the final model.

### 3.0 The Furnace Thermal Model

The model is arranged from the center out as follows. (See Figure D-2).

#### 3.1 Sample

The standard or nominal sample used for this analysis is 5cm diameter x 5cm long and pure iron. This shape was used because of its simplicity. Additionally, the size and weight can be changed by changing only three variables, the radius, length and density. Iron was chosen because it melts slightly below the operating temperature of the 1600°C furnace.

#### 3.2 Shroud

The shroud acts as a thermal radiation barrier between the sample and heaters.

#### 3.3 Heater

The heater constitutes the only source of heat within the furnace. Heat is transferred from it by radiative and convective heat transfer.

THERMAL PROBLEM

THERMAL ELEMENT

BREAKDOWN

ELECTRICAL ANALOG

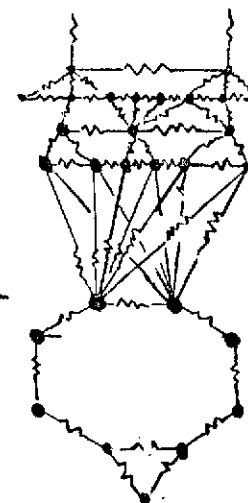
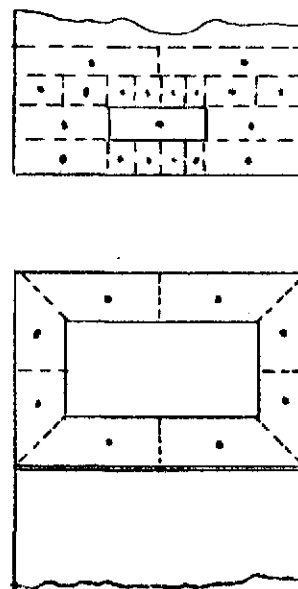
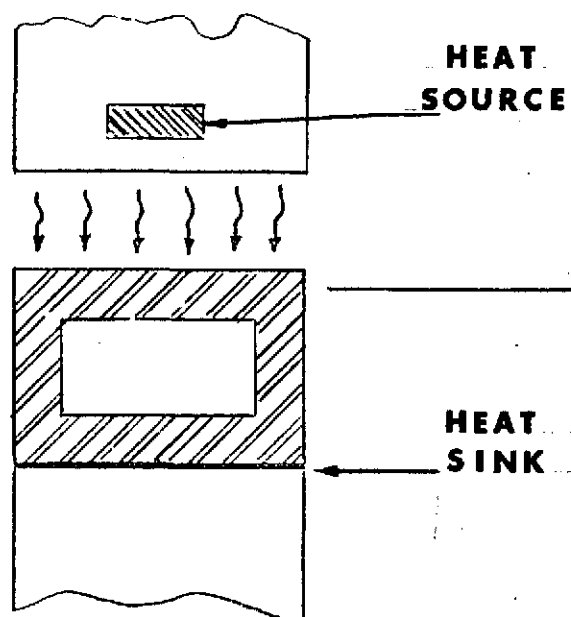


FIGURE D-1

THERMAL MODELING TECHNIQUE

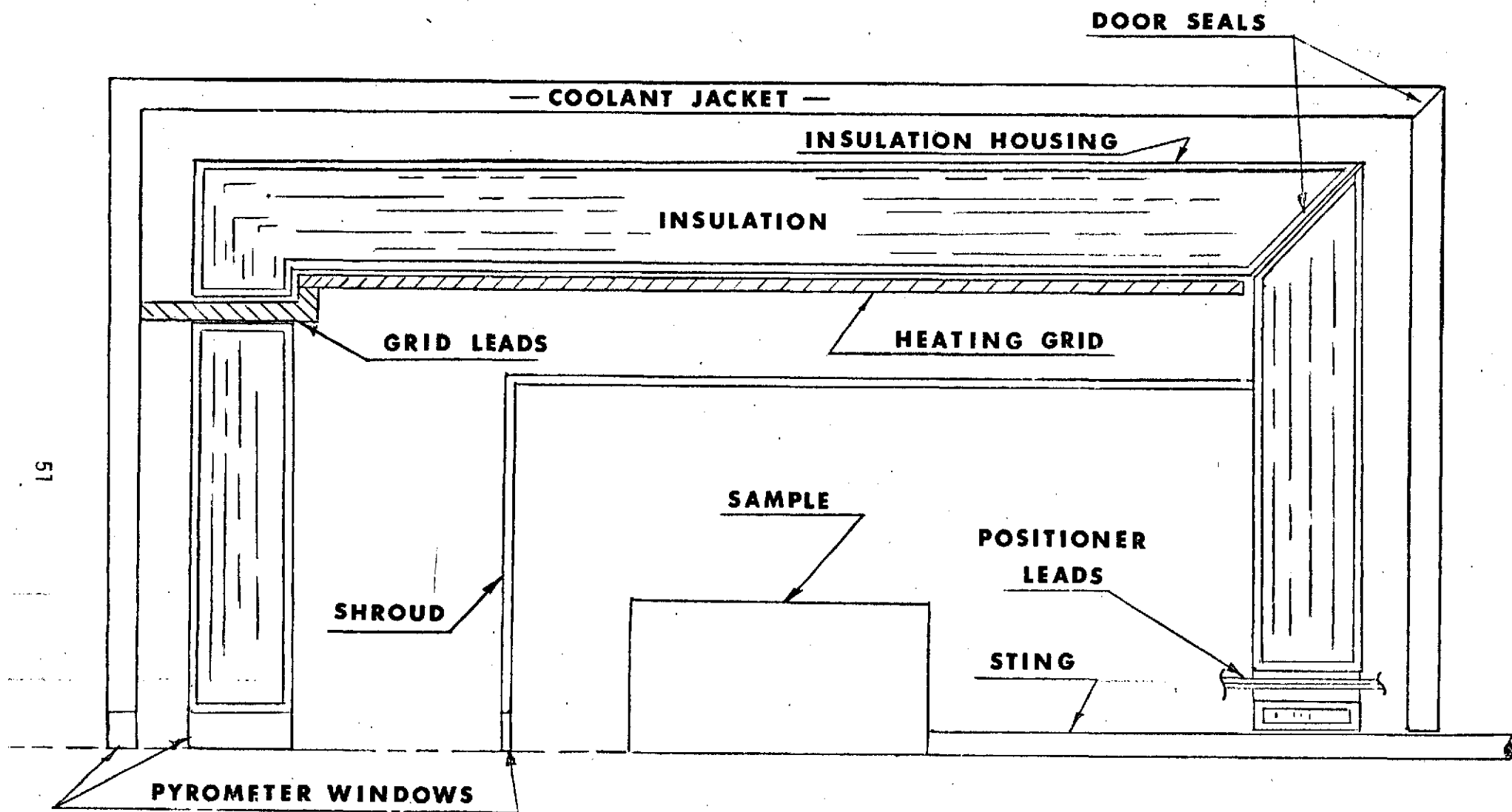


FIGURE D-2  
FURNACE SCHEMATIC

### 3.4 Insulation Housing

The structural housing surrounds the insulation package and represents the main mode of heat loss.

### 3.5 Insulation

Manufacturer's data indicate that the total heat flux through the insulation will be approximately 100 watts for the small furnace and 300 watts for the large furnace. Since this represents a small percentage of the total losses (2%-3%), the insulation was assumed perfect. This allowed significant program simplification and greatly decreased the computing time. The error associated with this simplification is within the limit of program computational error.

### 3.6 Atmosphere

Under vacuum conditions, the only available modes of heat transfer are solid conduction and radiation. Addition of a gaseous test media will also allow gas conduction. However, since the furnace will be operated in a weightless state, no gaseous convection can occur. The heat capacity of the gas is nil and it was therefore not included.

### 3.7 Heat Sink

Any heat which passes through the insulation or its housing must be absorbed to prevent an increase in temperature of the outside furnace surfaces. This is accomplished by the cooling fluid in the cooling jacket. The jacket is maintained at 41°C (105°F) by sufficient coolant flow.

### 3.8 Sample Support Sting

Samples may be supported by a sting. The sting, if used, is a heat path through the insulation.

### 3.9 Positioner Leads

Electric or magnetic field sample positioning elements must be powered and controlled. The leads are heat loss paths through the insulation.

### 3.10 Heater Leads

The high power requirements of the heater demand large heater leads. Since these must penetrate the insulation, they are heat loss paths.

### 3.11 Window

Some experiments may require a direct view of the sample. A quartz window to allow such viewing will allow radiative heat loss.

## 4.0 Furnace Geometry, Materials, Weights

The geometric and material properties of the furnace including parameter vibrations are presented below: See Appendix A for a summary of material properties.

### 4.1 Sample

- 4.1.1 Material - Iron for all cases
- 4.1.2 Shape - Cylindrical for all cases
- 4.1.3 Size - Nominal 5cm dia. x 5cm long  
Variation "A" 8.95cm dia. x 8.95cm long
- 4.1.4 Mass - Nominal .788 Kg (1.737 lb)  
Variation 4.1.3A 4.54 Kg (10.0 lb)
- 4.1.5 Position - Concentric with the furnace for all cases.  
Centered within the shroud for all cases, except the gradient runs.

### 4.2 Shroud

- 4.2.1 Material - Nominal Molybdenum  
Variation "A" Tantalum  
Variation "B" Tungsten  
Variation "C" Ceramic ( $ZrO_2$ ,  $ThO_2$ , etc.)
- 4.2.2 Shape - Nominal Spherical  
Variation 4.2.1C Cylindrical
- 4.2.3 Size - Nominal 17.78cm O.D. (Sph) .5cm tk.  
Variation "A" 25.4cm O.D. (Sph) .5cm tk.
- 4.2.4 Mass - Nominal .5 Kg  
Variations 4.2.1A .81 Kg  
4.2.1B .94 Kg  
4.2.1C 2 Kg
- 4.2.5 Position - Concentric with the furnace for all cases.

#### 4.3 Heater - 3 Phase Self-supporting Screen

- |       |                                |   |   |  |
|-------|--------------------------------|---|---|--|
| 4.3.1 | Material                       | - | Nominal<br>Variation "A"<br>Variation "B"                             | Molybdenum<br>Tantalum<br>Tungsten                     |
| 4.3.2 | Shape                          | - | Cylindrical   |  |
| 4.3.3 | Size                           | - | Nominal<br>Variation "A"  | 21.8cm O.D. x 20.8cm long<br>29.5cm O.D. x 27.9cm long |
| 4.3.4 | Mass                           | - | Nominal<br>Variation 4.3.3A   | ~2.75 Kg<br>~4 Kg                                      |
| 4.3.5 | Position                       | - | Concentric to the furnace, just within the insulation housing cavity. |  |
| 4.3.6 | Commercial Supplier - Sylvania |   |   |  |

#### 4.4 Insulation Housing

- |       |                            |   |   |  |
|-------|----------------------------|---|---|--|
| 4.4.1 | Material                   | - | Nominal<br>Variation "A"<br>Variation "B"                                   | Molybdenum<br>Tantalum<br>Tungsten   |
| 4.4.2 | Shape                      | - | Double walled cylinder. To enclose the insulation. Hermetic welds required. |  |
| 4.4.3 | Size<br>(Approximate)      | - | Nominal<br>Variation "A"<br>Variation "B"<br>Variation "C"                  | .5mm thickness to fit small furnace insulation.<br>.5mm thickness to fit large furnace insulation.<br>1.27mm thickness to fit small furnace.<br>1.27mm thickness to fit large furnace. |
| 4.4.4 | Mass                       | - | Nominal<br>Variation 4.4.1A<br>Variation 4.4.1B<br>Variation 4.4.1C         | ~2.57 Kg<br>~4.18 Kg<br>~4.85 Kg<br>~7.9 Kg  |
| 4.4.5 | Position                   | - | Concentric with furnace and totally enclosing the insulation.               |  |
| 4.4.6 | Commercial Supplier - TECO |   |   |  |

#### 4.5 Insulation

- 4.5.1 Type - Multi-foil, vacuum operation.
- 4.5.2 Material - Nominal Molybdenum  
Variation "A" Tantalum  
Variation "B" Tungsten
- 4.5.3 Shape - Hollow Cylinder
- 4.5.4 Size - Nominal 40-80 Layers, Est.  
2.5cm thick
- 4.5.5 Mass - Nominal 2.4 Kg
- 4.5.6 Position - Concentric with the furnace and totally enclosing the sample, shroud and heaters.
- 4.5.7 Commercial Supplier - TECO

#### 4.6 Atmosphere

- 4.6.1 Type - Nominal Vacuum  
Variation "A" Argon  
Variation "B" Hydrogen  
Variation "C" Oxygen
- 4.6.2 Pressure - Nominal 0.0 Atm  
Variation "A" .3 Atm  
Variation "B" 1.0 Atm

#### 4.7 Heat Sink

- 4.7.1 Material - Aluminum alloy in all cases.
- 4.7.2 Shape - Closed cylinder with cooling coils.
- 4.7.3 Size - Nominal 29.53cm dia. x 28.58cm long  
Variation "A" 37.15cm dia. x 36.2 cm long
- 4.7.4 Mass - Nominal 2.7 Kg  
Variation 4.7.3A 4.1 Kg
- 4.7.5 Position - Concentric to the furnace and totally enclosing all other parts.

#### 4.8 Support Sting

- 4.8.1 Material - Nominal Molybdenum  
Variation "A" Tantalum  
Variation "B" Tungsten  
Variation "C" Ceramic ( $ZrO_2$ ,  $ThO_2$ , etc.)
- 4.8.2 Shape - Tube
- 4.8.3 Size - ~.65cm dia. x 15-30cm long
- 4.8.4 Mass - < .2 Kg
- 4.8.5 Position - Mounted on door, concentric with the furnace.

#### 4.9 Window

- 4.9.1 Material - Fused silica
- 4.9.2 Shape - To fit metal shroud.
- 4.9.3 Size - 2.5cm dia. x .3cm thick
- 4.9.4 Mass - Negligible
- 4.9.5 Position - To be determined.



## 5.0 Summary of Thermal Assumptions

It was necessary to make some simplifying assumptions concerning thermal aspects of the furnace to minimize computer runs. These assumptions are listed below:

### 5.1 Perfect Insulation

The vacuum multi-foil insulation is very efficient thermally. Manufacturer's data indicate that total heat losses through the insulation including corner losses should be about 100 watts for the small 1600°C furnace and 300 watts for the large 2300° furnace. (Ref. D-1). These losses are so low compared to other system losses that perfect insulation was assumed. This assumption substantially reduced the computer time required to solve problems. The heat loss values quoted above may be added to the final results, if desired. However, the incurred error of <3%, if they are not added, is less than the expected maximum mathematical error.

### 5.2 Material Properties

Values of thermal conductivity, heat capacity, heat of fusion, and total normal emissivity were assumed constant, where possible, or as linear function of temperature, where necessary. These approximations, coupled with the inherent material property and structural variations of a manufactured furnace, result in anticipated calculational accuracy of approximately  $\pm 10\%$  for temperature and heat flux based upon results for similar thermal analyses.

### 5.3 Non-Deforming Sample

The standard sample chosen for the analysis is pure iron. Its selection was based upon its melt temperature of 1536°C (2797°F). During the melt phase, the sample will gradually change its shape from cylindrical to spherical. It is assumed that this shape change will not substantially affect the thermal response of the sample. It is estimated that the maximum increase in time to reach the desired temperature after sample melt will be approximately 14% due to a small decrease in the surface to volume ratio as the sample melts.

### 5.4 Ideal Heater

It has been assumed that the heater will be under full power from the start of the tests until the desired sample temperature is attained. Realistically, it may be necessary and advantageous to program the heater to decrease specimen thermal shock and improve the test power profile. In addition, it may be desirable

to trim the heater output as the sample nears the desired temperature. This would diminish the degree of overshoot and also decrease the peak heater temperature. However, both of these modifications will tend to slightly increase the time required to attain the desired sample temperature.

### 5.5 Ideal Heat Sink

It is assumed that the cooling jacket will be maintained at 41°C (105°F) during all experiments. Significant variations (i.e.  $\pm 100\%$ ) of this temperature possibly caused by emergency operation will not affect furnace response substantially.

## 6.0 Thermal Results

A parametric analysis of the furnace was conducted in order to determine the thermal sensitivity of the design to reasonable variations in furnace materials, dimensions, test atmosphere, and sample size. All results are based on a so-called "Nominal" furnace and "Nominal" experiment unless otherwise indicated. A description of these nominal conditions is presented in Table D-1. The results of the analysis are presented in Figures D-3 through D-13. The three most important dependent variables are furnace component temperature, time for sample to attain temperature, and steady state furnace heat loss.

Figure D-3 presents the temperature history of the nominal furnace sample from start-up at ambient temperature (assumed 41°C) to steady state at 1600°C. Figure D-4 presents a combination of sample temperature histories for vacuum, argon, and hydrogen atmosphere operation. An important observation is that the presence of a gas in the furnace does not significantly perturb the thermal response of the sample. Figure D-5 presents the temperature history for the large furnace design. Figure D-6 is a parametric comparison of the steady state heat losses from the small and large furnaces for 1600°C and 2300°C experiments. Figure D-7 presents the time required for the sample to attain 1600°C as a function of the type of atmosphere. This is interesting in that the heat-up time is minimum for a nitrogen parametric atmosphere. This result is plausible when considering that the vacuum case is most efficient in deterring heat loss and the most inefficient for transferring heat to the sample. At the other extreme, this situation reverses itself. The hydrogen transfers heat to the sample but also causes a greater heat loss. As a result, the best atmospheric condition for sample heating is between these two extremes. Figure D-8 presents this heat loss as a function of the conductivity of the test atmosphere (at 1600°C). The asymptotic nature of this curve with increasing gas conductivity is to be expected. At approximately the conductivity of helium, the temperatures in the

furnace cavity are essentially uniform. Any further increase in gas conductivity is inconsequential since no significant temperature gradients exist.

Some of the experiments being considered require that a temperature gradient be established and maintained on a long cylindrical specimen. This is used to measure the grain-growth response of the sample to a temperature gradient. The gradient is produced by drawing the heated sample against the furnace door which is subsequently flooded with helium. This cools the inner surface of the door. If the furnace heater remains on, a steady temperature gradient will be imposed on the specimen. Figure D-9 presents the response of an iron sample .635 cm in diameter and 7.62 cm long in such a test. A steady temperature gradient is produced in approximately .1 hour.

Variations in the magnitude of such a gradient can be readily accomplished by use of different heater power settings, the positioning of radiation shields, and through design of the heat sink in the door.

Figure D-10 presents the time required to heat the sample to 1600°C versus the sample weight. At sample weights above approximately 1.0 Kg, the time required to attain 1600°C operation is essentially linearly related to sample weight. The zero-sample time is the time required for the heating element to attain 1600°C and represents the lower limiting time for a very small sample.

The temperature distributions within the furnace for some of the extreme conditions analyzed are presented in Figures D-11 through D-13.

A summary of steady state heat losses from the various furnace options considered is presented in Table D-2. The lowest loss of 2.9 Kw is experienced with the small furnace operating at 1600°C in vacuum. The highest loss of 6.3 Kw is experienced with the large furnace operating at 2300°C in hydrogen.

The heat loss values are based on conservative assumptions. However, several methods of reduction do exist, the following appear most promising:

#### 1. Longer Door Seal Heat Paths

Thermal energy passes readily through the door edges. These edges are thick since they must be capable of supporting pressure loads during some experiments. Heat losses could be reduced by increasing the effective length of the door edge. The savings will be approximately proportional to the length increase. Doubling this length will reduce the door losses

by approximately 50% (equivalent to an over-all heat loss reduction of 30% or .9 Kw). It must be reemphasized that any door edge configuration must also be capable of structurally supporting the insulation housing during operation.

2. Small Furnace Diameter

Heat losses can be substantially reduced by decreasing the furnace diameter. The heat loss through the door edges should be proportional to the square of the diameter. Therefore, a 30% reduction in furnace diameter should produce a 50% reduction in door heat losses (30% overall).

3. Reduction of Heater Lead Size

The cross sectional area of the heater leads can be reduced somewhat to allow a reduction of heat losses.

TABLE D-1

NOMINAL FURNACE EXPERIMENT PARAMETERS

Size . . . . .	.0195m <sup>3</sup> ("Small" furnace)
Weight . . . . .	< 12 Kg
Material . . . . .	Molybdenum alloy
Max. Operating Temp. . . . .	1600°C
Test Atmosphere . . . . .	Vacuum
Test Sample . . . . .	Iron cylinder 5cm dia. x 5cm long (wt - .79 Kg)
Start-Up Power . . . . .	6 Kw to 1600°C
Sustained Power . . . . .	Dependent variable approximately 3 Kw, average

FIGURE D-3  
SAMPLE TEMPERATURE  
VS.  
TIME

6 KW FOR .168 HRS

6 KW FOR .1 HRS THEN 3.25 KW

ALL CURVES ARE FOR THE NOMINAL  
FURNACE AT NOMINAL CONDITIONS  
UNLESS OTHERWISE INDICATED.

TEMPERATURE - °C

TIME - HOURS

FIGURE D-4  
SAMPLE TEMPERATURE  
VS.  
TIME  
FOR VARIOUS TEST ATMOSPHERES

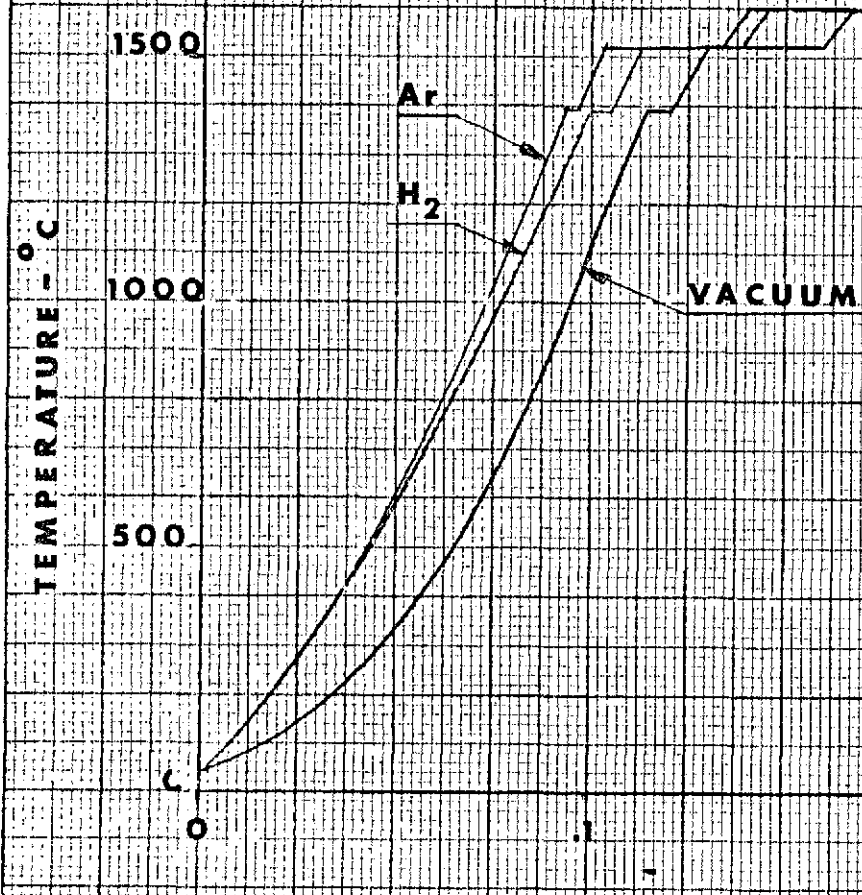


FIGURE D-5

SAMPLE TEMPERATURE  
VS.  
TIME

LARGE FURNACE, 9 KW. INPUT TO 2300 °C

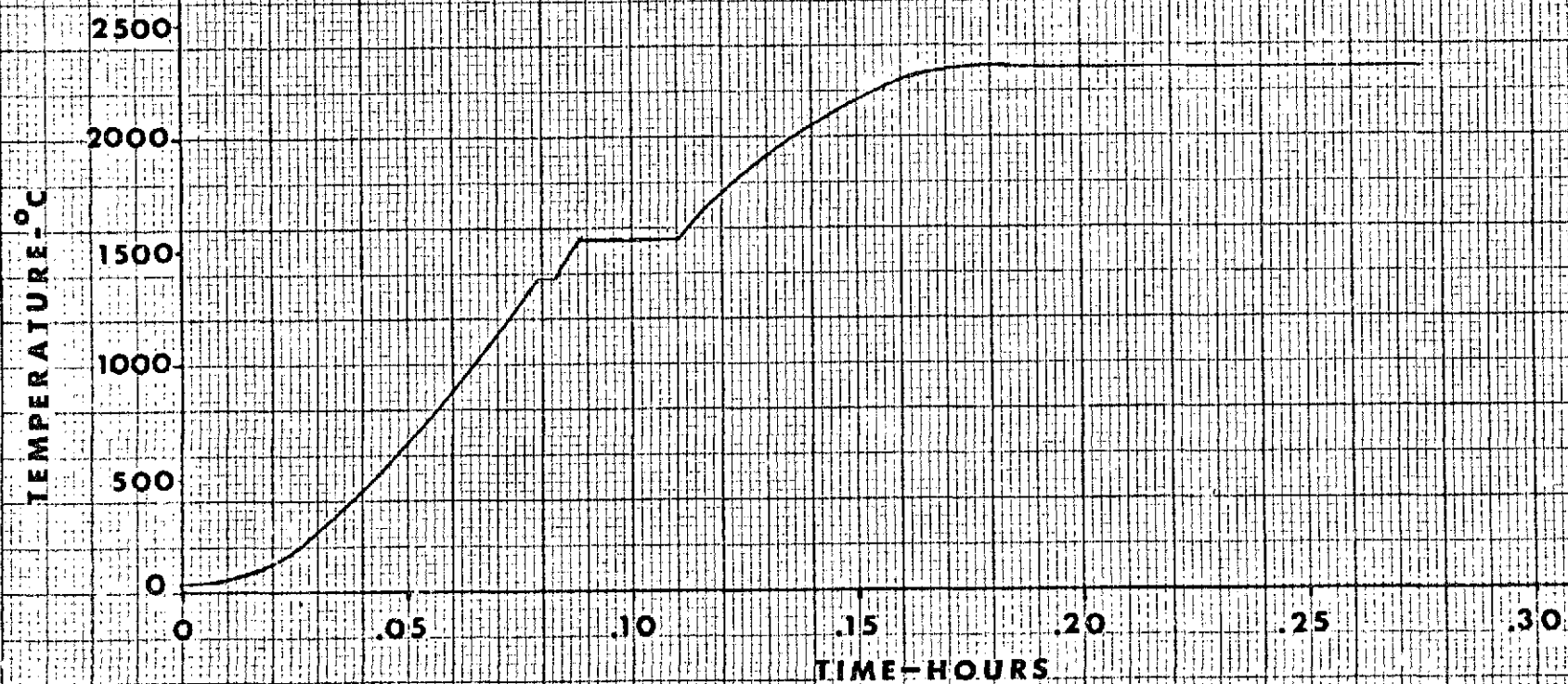




FIGURE D-6

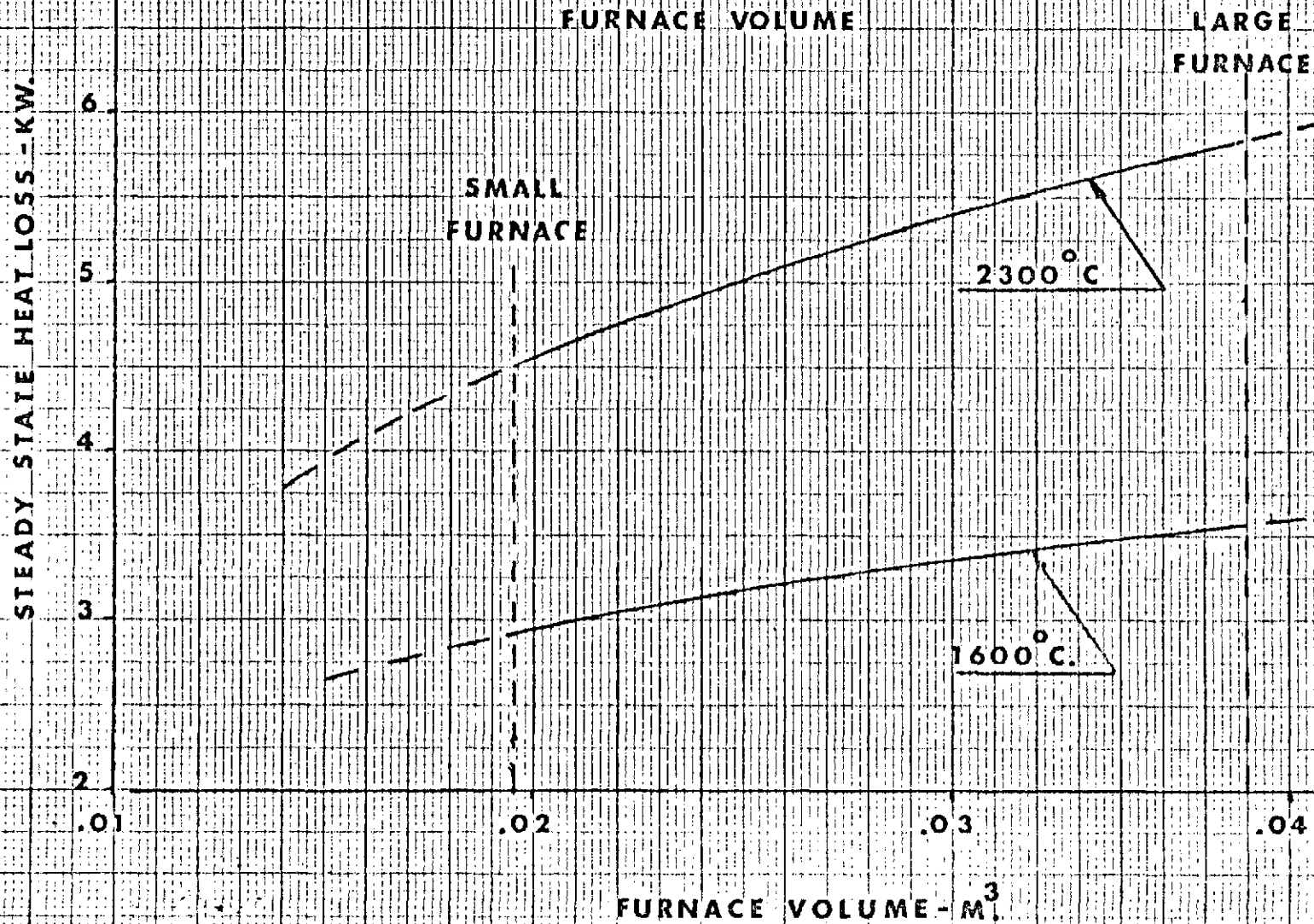
STEADY STATE HEAT LOSSES  
VS  
FURNACE VOLUME

FIGURE D-7  
TIME FOR 1600°C. SAMPLE  
VS.  
GAS CONDUCTIVITY

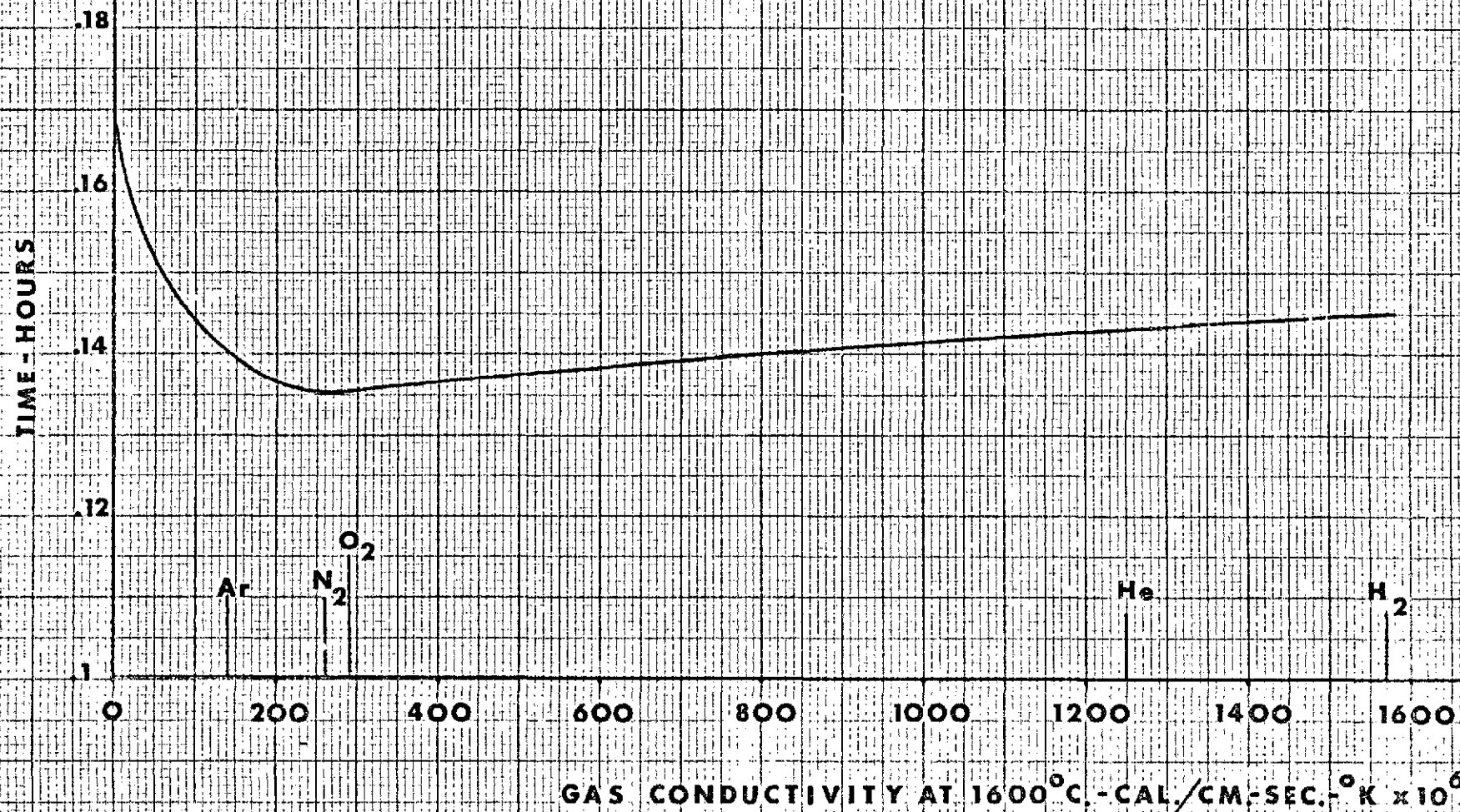


FIGURE D-8  
STEADY STATE HEAT LOSS  
VS  
GAS CONDUCTIVITY

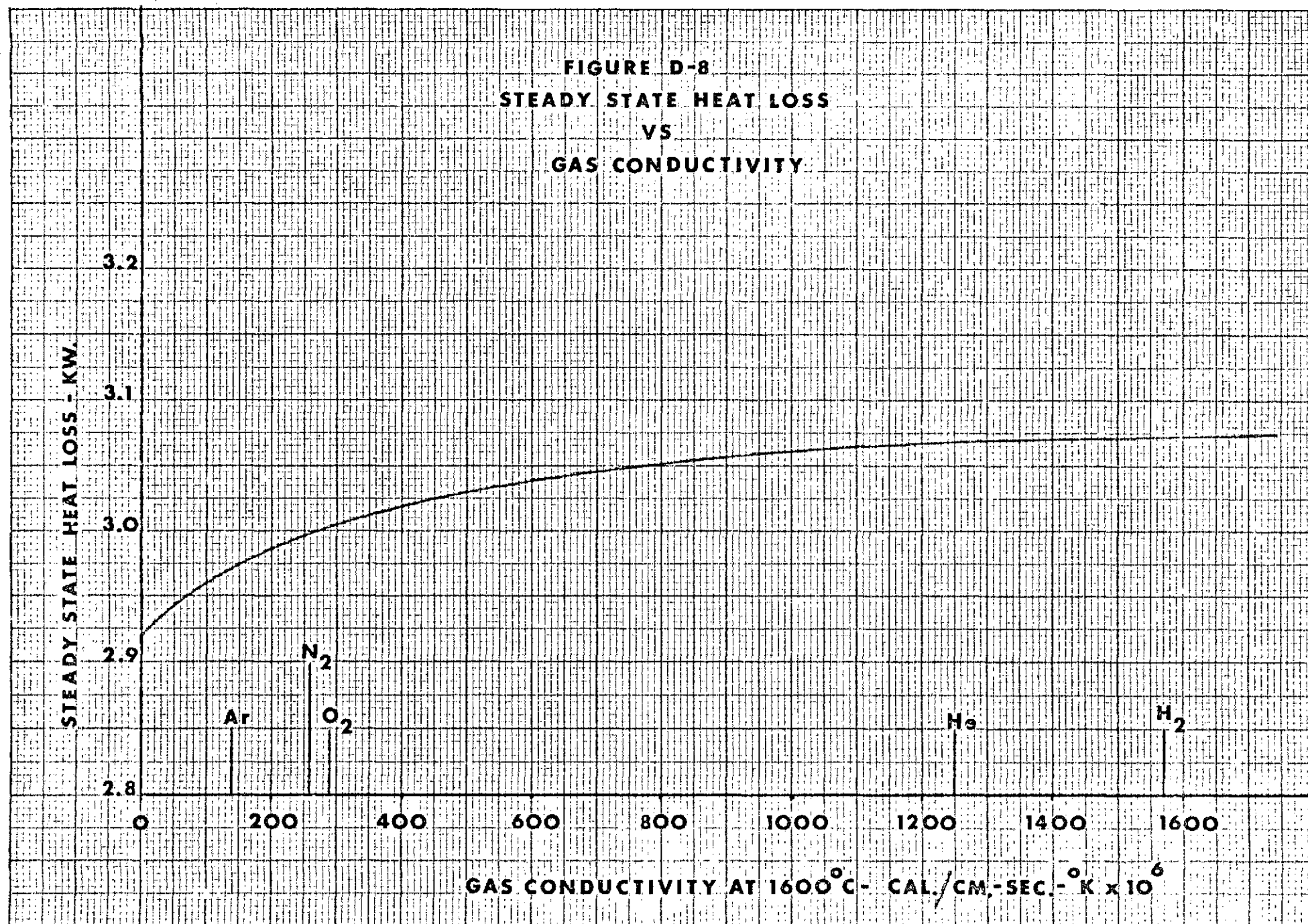
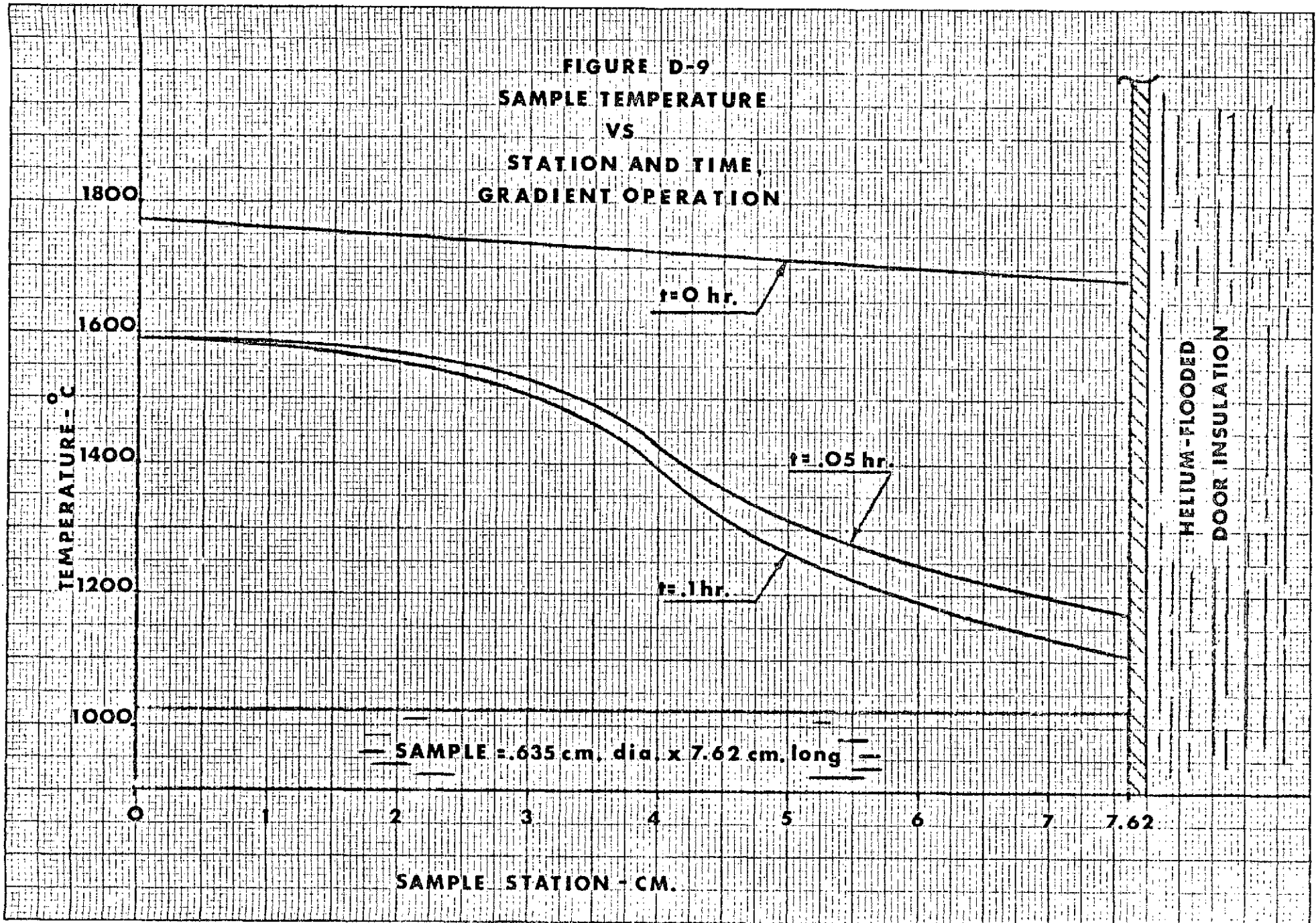
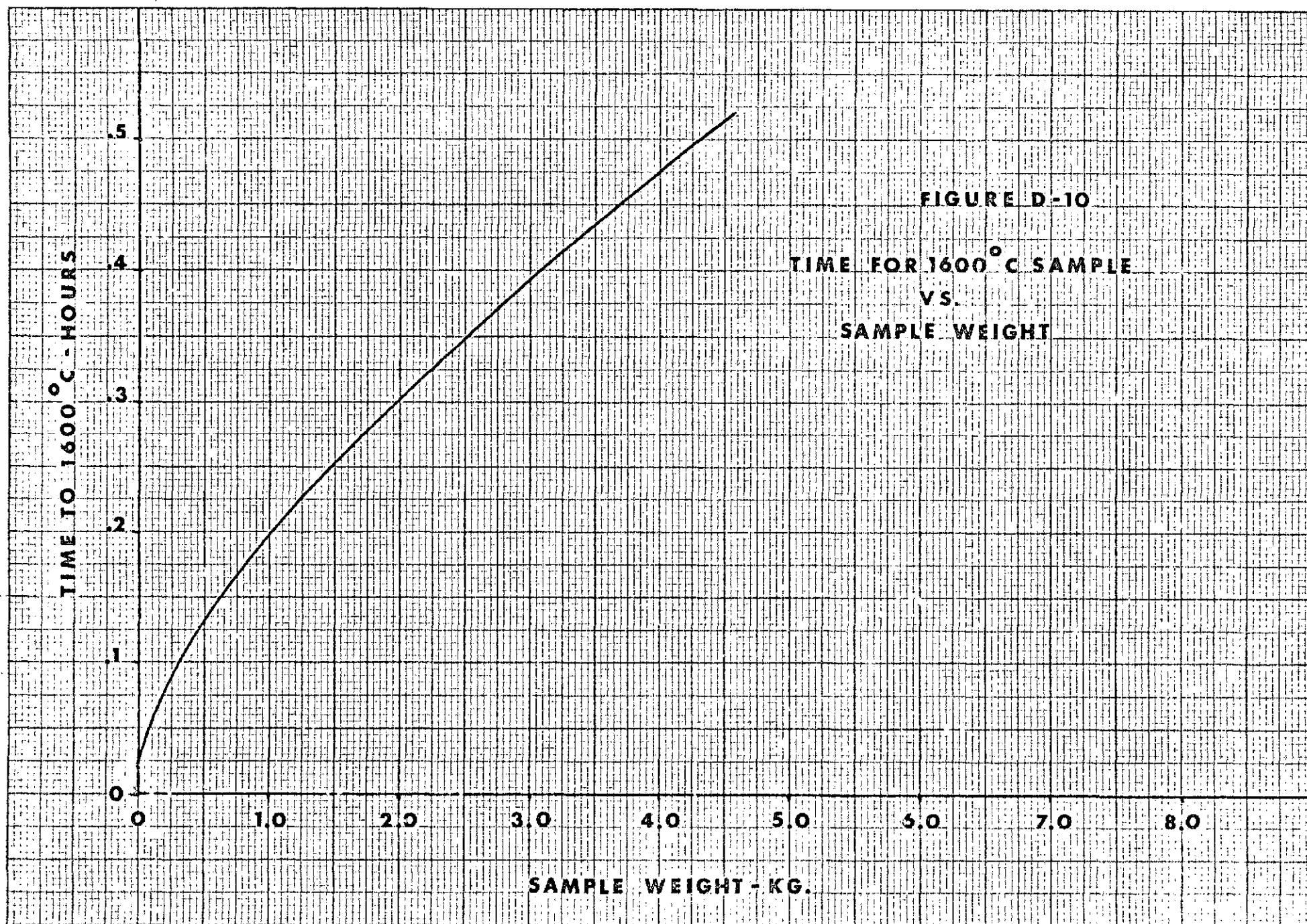


FIGURE D-9  
SAMPLE TEMPERATURE  
VS  
STATION AND TIME,  
GRADIENT OPERATION







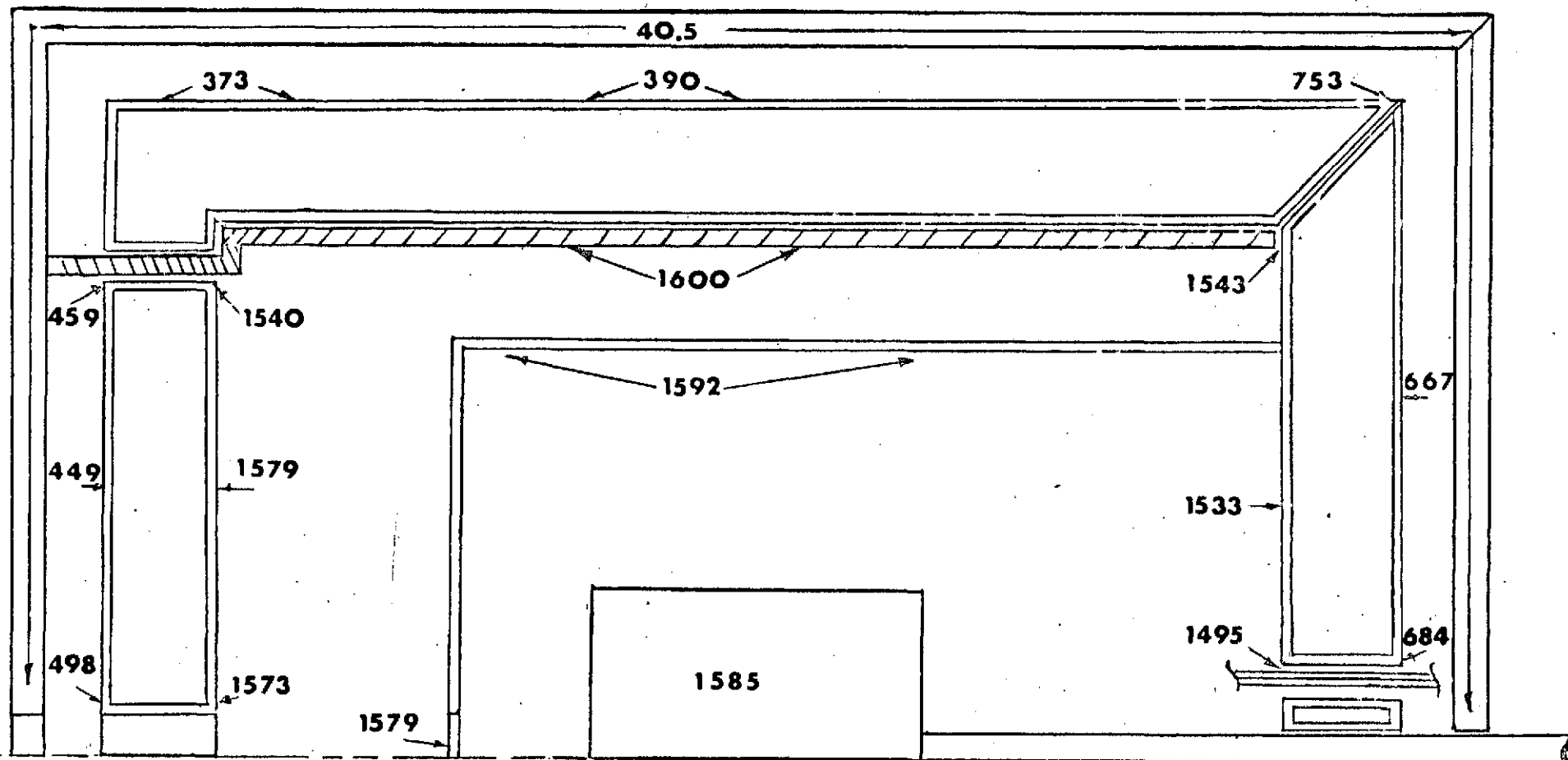


FIGURE D-11  
FURNACE TEMPERATURE DISTRIBUTION  
IN VACUUM- $^{\circ}\text{C}$  ( $1600^{\circ}\text{C}$ )

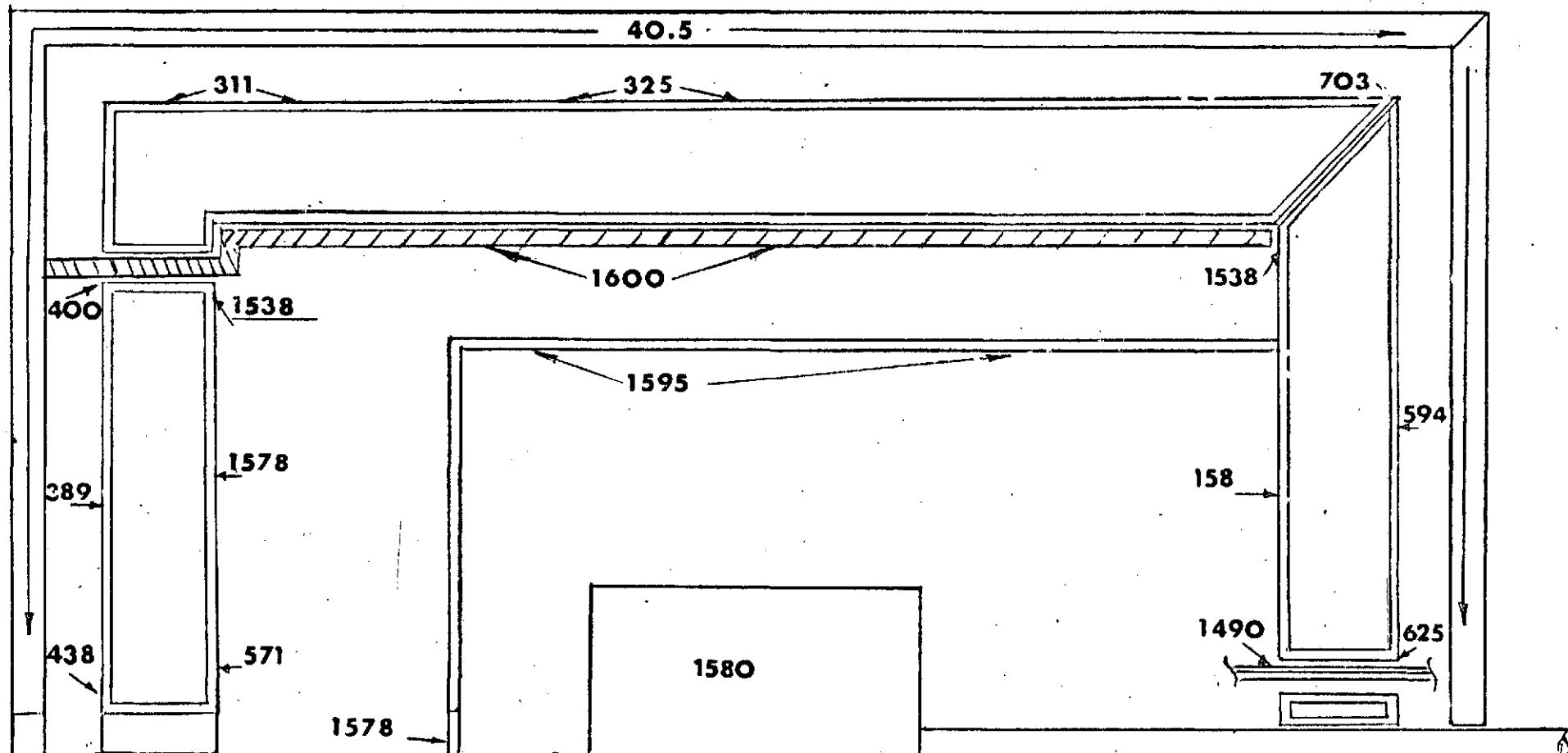


FIGURE D-12  
FURNACE TEMPERATURE DISTRIBUTION  
IN HYDROGEN-°C (1600°C)





TABLE D-2

FURNACE STEADY STATE HEAT LOSSES

Conditions	Heat Losses (Watts)					
	Insulation Housing	Door Seal	Power and Control Lines	Sample Support Sting	Window	Total
1. Small furnace at 1600°C, Vacuum	584	1745	495	55	27	2906
2. Small furnace at 1600°C, Hydrogen	625	1868	495	55	27	3070
3. Small furnace at 2300°C, Vacuum	905	2750	748	84	72	4559
4. Small furnace at 2300°C, Hydrogen	968	2940	748	84	72	4812
5. Large furnace at 1600°C, Vacuum	652	2300	528	58	20	3558
6. Large furnace at 1600°C, Hydrogen	698	2461	528	58	20	3765
7. Large furnace at 2300°C, Vacuum	1075	3979	756	84	72	5966
8. Large furnace at 2300°C, Hydrogen	1150	4260	756	84	72	6322

## APPENDIX E

### Components

The heating elements and the insulation package are two key components in the presented concept. Heating element design is based on using wire mesh elements while insulation design is based on using multi-foil insulation.

Conventional high temperature furnace heating elements are made of sheet materials, usually molybdenum or tungsten. The thin sheets are formed into cylinders and connected, as a resistance heating element to a suitable power supply. The sheet elements have a tendency to warp and become distorted as a result of the heating and cooling cycles. Sheet elements have a limited life and fail by cracking.

An advanced design in commercially available high temperature heating elements is structured around the use of free hanging wire mesh elements. The wire in the mesh heating elements can be manufactured to closer tolerances than sheet materials and hence, possess more uniform resistance with resultant minimization of temperature gradients. Wire mesh element designs lend themselves to longer operating life with estimates of ten times that of conventional sheet type elements.

Traditionally, high temperature vacuum furnace designs utilized bulky sheet type radiation shields to act as thermal barriers. Under sponsorship of the United States Atomic Energy Commission there has been developed a concept known as multi-foil thermal insulation for high temperature vacuum applications. Multi-foil thermal insulation consists of many layers of thin metal foil separated from each other by high purity refractory oxide particles. The layers of metal foil, which typically range from .0063mm to .025mm thick, act as thermal radiation barriers. The oxide particles prevent adjacent layers of metal foil from coming in contact with one another to form a metal-to-metal conduction path away from the source of heat. The oxide particles are a few microns in diameter and are sprayed onto one side of each foil layer. The particle coatings are relatively sparse and the low thermal conductivities of the oxides, plus the high contact resistances that exist in vacuum between particles and foil, combine to minimize the conduction component of the total heat transfer through the insulation. Typically, the thermal conductivity of this type of insulation operating in a vacuum is exceptionally low ranging from  $10^{-7}$  watts/cm-°K to  $10^{-5}$  watts/cm-°K, depending on the temperature level and the particular materials involved.

Multi-foil insulation is available commercially for temperatures up to 3000°C. For temperatures of 2400°C, insulation of tungsten or molybdenum foils with  $ZrO_2$  particles is available.